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Total Exposure Assessment Methodology (TEAM) Study: Standard Operating Procedures Employed in Support of an Exposure Assessment Study
Volume 4

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June 1987

THE TOTAL EXPOSURE ASSESSMENT METHODOLOGY (TEAM) STUDY: STANDARD OPERATING PROCEDURES EMPLOYED IN SUPPORT OF AN EXPOSURE ASSESSMENT STUDY

VOLUME IV

by

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16. ABSTRACT

The TEAM Study measured exposures to 20-25 volatile organic compounds in the air, drinking water, and exhaled breath of 650 persons in 4 states. Volume I is a summary and overview of the entire study. Volume II deals with studies in New Jersey, North Carolina, and North Dakota. Volume III deals with studies in California. Volume IV presents the Standard Operations Procedures employed in the study.

Major findings include: 1) personal monitors and breath spirometers employing Tenax adsorbents are sensitive and adequately precise instruments to determine normal daily exposures of the general public; 2) personal and indoor exposures generally exceeded outdoor concentrations; 3) major sources of exposure include occupations, smoking, visiting dry cleaners, and filling gas tanks.

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PREFACE

This volume contains a set of standard operating procedures (SOPs) which were implemented in support of the Total Exposure Assessment Methodology (TEAM) Study. They may also be employed for future exposure studies. The SOPs cover activities relevant to the chemical sampling and analysis for volatile organic chemicals in personal air, fixed-site air, human breath and drinking water samples. They also address the preparation of data files on chemical levels for subsequent statistical analysis.

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RTI/ACS-SOP-120-001

November 1983

Revision 0

Standard Operating Procedure for the Proper Use and Maintenance of Laboratory Notebooks

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Date

Standard Operating Procedure for the Proper Use and Maintenance of Laboratory Notebooks

RTI/ACS-SOP-120-001

1.0 INTRODUCTION

The purpose of laboratory notebooks is to provide a permanent legal record of experimental procedures and results. Thus, the laboratory notebook serves as the principal means for documenting and evaluating project performance and data quality.

2.0 SCOPE OF PROCEDURE

This standard operating procedure describes in detail the rules and regulations which govern the proper use and maintenance of laboratory notebooks in the performance of all ACS projects.

3.0 LABORATORY NOTEBOOK RULES AND REGULATIONS

3.1 Notebook Assignments

3.1.1 Request Notebooks by dialing x6210 (Hanes Building). Give name of individual to whom the Notebook should be issued and the RTI project number for which data will be recorded.

NOTE: The Notebook should be issued to the chemist performing the work and not to the Project Leader or supervisor.

3.1.2 Request and use a different Notebook for each assigned project.

NOTE: If the staff member is involved in several concurrent, low level-of-effort programs (e.g., projects involving commercial clients who periodically submit analytical requests), the experimental work performed on these projects may be described in the same notebook. Consult with supervisor.

3.1.3 Issue a Notebook to all staff members assigned to work on a specific project.

NOTE: Staff members who are assigned a temporary, short-term task may record that data in the project Notebook issued to a co-worker. Consult with supervisor.

3.2 Recording of Data

3.2.1 All project work which results in the generation of data must be recorded in the Notebook of the chemist who performed the work.

NOTE: If a team of individuals are assigned to a common task, the project activity may be recorded either in a separate project Notebook issued for that purpose or in one of the co-workers Notebooks. In either case, the participating analysts must sign and date each entry. Consult with supervisor.

3.2.2 All pages must be dated and signed on the day of recording data.

3.2.3 All entries must be recorded in black ink.

3.2.4 All entries must be legible and written in understandable prose.

3.2.5 All entries must be written directly in the Notebook and not on separate pieces of paper to be copied later.

NOTE: In the event that it is not possible or practical to record data directly into the Notebook, write the data on a suitable surface and then permanently attach this material to the proper Notebook page as soon as possible.

3.2.6 All entries must be recorded promptly; i.e., describe project activity in writing soon after its completion.

NOTE: This must be the day that you perform the work. Do not trust your memory.

3.2.7 All complete and partial pages not used in recording data must be voided by placing an "X" or "Z" on the unused portion of the page.

3.2.8 Start and maintain a Table of Contents to facilitate retrieval of specific project data. Update the Table of Contents after every entry.

3.2.9 Witnessing data entries is not necessary unless the Project Director or QA Officer requires this action.

3.2.10 If project Notebook contains confidential data, copying pages should be kept to a minimum or strictly controlled. Consult with supervisor.

3.2.11 Staff members assigned to off-site project duties (e.g, sample collection) will not need to record their daily activity in a laboratory Notebook. The Project or Task Leader will identify project staff assigned to off-site duty and their schedule by memo to the QA Officer.

3.3 Notebook Storage

3.3.1 All Notebooks must be easily accessible; e.g., on a desk or bookshelf.

NOTE: If a Notebook contains confidential data for a commercial client or for a program requiring security, a special Notebook storage area will be designated by the Project Leader.

3.3.2 Do not remove the Notebook from the building unless it is necessary to document a legitimate off-site project activity.

NOTE: The use of a Notebook for this purpose must be approved by the Project Leader.

3.4 Notebook Errors

3.4.1 Any errors or invalid data in the Notebook must be so designated by drawing a single line through the entry. The individual making this notation must initial the action and provide an explanation for the alteration, if appropriate.

3.4.2 If errors or omissions are noted at a later date, record the correction on the page currently being used and reference the page in error. On the latter page, indicate error and reference page with corrected entry.

3.5 Completed Notebooks

All laboratory Notebooks contain project raw data and must be retained in a safe and secure location after they are full. At this point, they are technically part of the Analytical and Chemical Sciences (ACS) archives.

Regardless of who assumes custody of the completed Notebooks, the QA Office must keep a record of their location.

3.5.1 When the project has been completed, return the Notebook(s) to the Project Leader or your supervisor.

3.5.2 When a Notebook is full, retain it for reference and request a new one.

3.5.3 Upon termination of an employee's employment at RTI, all Notebooks assigned to the employee shall be transferred to the Office of the Vice-President.

NOTE: Approval for release of the final paycheck will be subject to this transfer; an assessment is made by the Office of Personnel during the termination interview that notebooks, building keys among other items have been properly transferred or returned.

3.6 Lost Notebooks

3.6.1 If a Notebook has been misplaced, damaged, or lost, notify your supervisor (or Project Leader) who will inform the QA Officer, investigate the incident, and report in writing to the Offices of Personnel and of the Vice-President.

3.7 Responsibility

3.7.1 The individual to whom the Notebook was assigned shall be responsible for the safekeeping of the Notebook.

3.7.2 The supervisor (or Project Leader) shall be responsible for informing the QA Officer which laboratory Notebooks have been issued for each project and to whom they were assigned.

3.7.3 The QA Officer shall be responsible for periodically monitoring and assessing ACS staff adherence to the regulations described in this SOP and submitting a report to the Vice-President. In addition, unannounced checks on upkeep of Notebooks may be conducted by the Vice-President.

3.7.4 The Vice-President shall be ultimately responsible for ensuring the proper maintenance of laboratory Notebooks.

4.0 OUTLINE OF A TYPICAL NOTEBOOK ENTRY

4.1 Place a heading (title) at the top of the page describing the nature of the work to be done.

4.2 Write a brief statement of purpose or intent of work.

4.2.1 Include pertinent Notebook and literature references.

4.3 Describe in detail the project activity performed.

4.3.1 Reference description of same procedure carried out previously only if the writeup is in the same Notebook presently being used. If the description is in another Notebook, the methodology must be described again.

4.3.2 Do not copy verbatim or paraphrase the procedure followed from a SOP, protocol, or other written document. Describe the operation performed in your own words.

4.3.3 Give particular attention to a description of the equipment and instrumentation used. Specify Model number, location, and instrument Notebook number.

4.3.4 Examples of experimental details which must be recorded:

4.3.4.1 Chemicals, reagents, and other materials used, identifying specific lot nos.

4.3.4.2 Gross weights and tares; identify balance used.

4.3.4.3 Preparation of calibration solutions.

4.3.4.4 Instrumental conditions used.

4.3.4.5 QA/QC measures followed.

NOTE: Emphasis is placed on specific details.

4.3.4.6 Results of calculations generated through the use of electronic or mechanical devices, e.g., calculators, computers, or sliderule.

4.4 Describe results of work performed.

4.4.1 Identify by coding and cross-referencing all original raw data that is not suitable for Notebook entry, e.g., spectra and computer printouts, and indicate the storage location for these items.

4.4.2 The data output described in 4.4.1 must be labelled with the Notebook number and page on which the laboratory procedure is described.

4.4.3 If copies of such data are put into Notebook, indicate where the original is located.

4.4.4 Record not only all observations which were a normal consequence of performing the work but also any unusual or unexpected behavior. The use of Tables and Summaries for presentation of results is encouraged.

4.5 Conclusions/Recommendations

Examples of topics suitable for discussion in this Section.

4.5.1 State conclusions as they relate to objectives of work.

4.5.2 Suggest ideas for future work.

4.5.3 Identify problem areas.

4.5.4 Indicate likely or recommended course of investigation, if applicable.

4.5.5 How will information or product obtained from this effort be utilized? For example, (1) the measured breakthrough volumes will be used in quantitating target compounds in breath samples or (2) Tenax cartridges with loaded standards will be processed by GC/MS.

5.0 REFERENCE

Laboratory Notebooks, RTI Policy and Procedures Manual, Memorandum No. 911.

RTI/ACS-SOP-120-002

January 1984

Revision 0

Standard Operating Procedure
for the Proper Use and Maintenance
# of Instrument Log Notebooks

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Officer
Date

Standard Operating Procedure for the Proper Use and Maintenance of Instrument Log Notebooks

RTI/ACS-SOP-120-002

1.0 INTRODUCTION

The purpose of instrument log notebooks is to provide a record of the maintenance, daily use, and current status of a particular laboratory instrument.

2.0 SCOPE OF PROCEDURE

This standard operating procedure describes in detail the guidelines which govern the proper use and maintenance of instrument log notebooks in the performance of all ACS projects.

3.0 INSTRUMENT LOG GUIDELINES

3.1 Notebook Assignments

3.1.1 Request Notebooks by dialing x6210 (Hanes Building). Give name of individual to whom the Notebook should be issued and the appropriate ACS overhead number for the laboratory in which the instrument is located.

NOTE: The Notebook should be issued to the individual responsible for the operation and upkeep of the instrument.

3.1.2 Request and use a different Notebook for each instrument.

NOTE: An instrument is defined as one from which analytical information is obtained, such as a GC, HPLC, UV, IR, AA.

Laboratory equipment such as balances and the pH meters have an impact upon analytical data, and also require an instrument log Notebook.

Laboratory equipment such as pumps and shakers are not considered analytical instruments but may be included as a part of an analytical system.

3.2 Recording of Data

3.2.1 Written records must be maintained of all inspection, maintenance, testing, calibrating, and/or standardizing operations. The record must show in sufficient detail the methods and materials used and the schedules used in routine inspection, cleaning, maintenance, testing, calibration/standardization of the equipment. Remedial action taken to correct instrument failure or malfunction shall be documented.

3.2.2 All pages must be dated and signed on the day of recording the entry.

3.2.3 All entries must be legible and written in understandable prose.

3.2.4 All entries must be recorded promptly; i.e., describe project activity in writing soon after its completion.

3.3 Notebook Storage

3.3.1 All Notebooks must be easily accessible; e.g., on a desk or bookshelf and proximate to the instrument.

3.3.2 Do not remove the Notebook from the building.

3.4 Notebook Errors

3.4.1 Any errors or invalid data in the Notebook must be so designated by drawing a single line through the entry. The individual making this notation must initial the action and provide an explanation for the alteration, if appropriate.

3.4.2 If errors or omissions are noted at a later date, record the correction on the page currently being used and reference the page in error. On the latter page, indicate error and reference page with corrected entry.

3.5 Completed Notebooks

All instrument log notebooks when full are technically part of the Analytical and Chemical Sciences (ACS) Archives.

Regardless of who assumes custody of the completed Notebooks, the QA Office must keep a record of their location.

3.5.1 When a Notebook is full, retain it for reference and request a new one.

3.5.2 When a Notebook is full, it may be kept with the instrument for reference or turned over to the QA Officer.

3.6 Lost Notebooks

3.6.1 If a Notebook has been misplaced, damaged, or lost, notify your supervisor (or Project Leader) who will inform the QA Officer, investigate the incident, and report in writing to the Office of Personnel and of the Vice-President.

3.7 Responsibility

3.7.1 The Notebook should be issued to the individual responsible for the operation and upkeep of the instrument.

3.7.2 The individual to whom the Notebook is assigned shall be responsible for the safekeeping of the Notebook.

3.7.3 The Notebook assignee shall be responsible for informing the QA Officer which laboratory Notebooks have been issued for each instrument.

3.7.4 The QA Officer shall be responsible for periodically monitoring and assessing ACS staff adherence to the regulations described in this SOP and submitting a report to the Vice-President. In addition, unannounced checks on upkeep of Notebooks may be conducted by the Vice-President.

3.7.5 The Vice-President shall be ultimately responsible for ensuring the proper maintenance of laboratory Notebooks.

4.0 OUTLINE OF A TYPICAL NOTEBOOK

The instrument log Notebook(s) shall contain four different types of information.

4.1 Analytical System

The first several pages of the Notebook shall be reserved for a description of the basic analytical system and all subsequent modifications.

4.1.1 Specify instrument model number, serial number and location. Instruments with component parts shall be identified by the major or most important component in the unit.

4.1.2 The component parts, configuation, and peripherals used shall be listed with their model numbers and serial numbers, if available.

4.1.3 Modifications to the system shall be included in this section. These would include addition of a module or detector, but not the changing of columns in a GC or HPLC.

4.1.4 An example of a typical status page is shown in Figure 1.

4.2 Daily Log

The purpose of the daily log is to maintain a running account of operational details.

4.2.1 An entry shall be made for each day of operation and should include the operator's name, the date, project and a description of the specific operating conditions of the day (temperatures, column, etc.). Comments on the type of samples run must be included (silyl derivatives, standards only, dirty biological samples, etc.).

4.2.2 If system performance runs are made or if a calibration is done the data shall be included or referenced. Calibration data which are appropriately included in a project notebook must be referenced in the instrument log. Other calibration data, test runs, or performance runs shall be recorded in the daily log section or in an accessible folder or binder which is referenced in the Notebook.

4.2.3 Example of a daily log entry is shown in Figure 2.

4.3 Sample Log

A record of all samples analyzed on an instrument shall be maintained.

4.3.1 The record of all analyzed samples may be included in the project notebook. In this case, the project notebook shall be referenced in instrument log notebook, Section 4.2.1.

4.3.2 The daily sample log may be part of the daily log section of the instrument log notebook (Section 4.2).

4.3.3 An example of a sample log entry is shown in Figure 3.

4.4 Instrument Maintenance Log

4.4.1 These records must contain the date of the operation and describe whether the maintenance operations were routine. Written records must be kept of nonroutine repairs performed on equipment as a result of instrument failure and malfunction. Such records must document the nature of the defect, how and when the defect was discovered, and any remedial action taken to correct the defect.

4.4.2 This information is best kept in the instrument log by working from the back of the Notebook toward the front.

4.4.3 Examples of instrument maintenance entries are shown in Figures 4 and 5.

6-25-82	Capillary plumbing installed for side A by PAH. (injector, splitter, septum purge, etc)
6-28-82	NPD installed on side A by MDW.
11-1-82	NPD removed from side A.
11-2-82	Detector base replaced by Joe Younger. Capillary pressure valve installed and capillary now operable on side A with FID. Side B is now packed column FID.
12-1-82	FPD installed on side B.

pneumatics: (note - air & H_2 lines must be switched at the air/H_2 block)

flows:
air #1: 80 ml/m
air #2: 170 "
H_2: 140 "
carrier: 30"

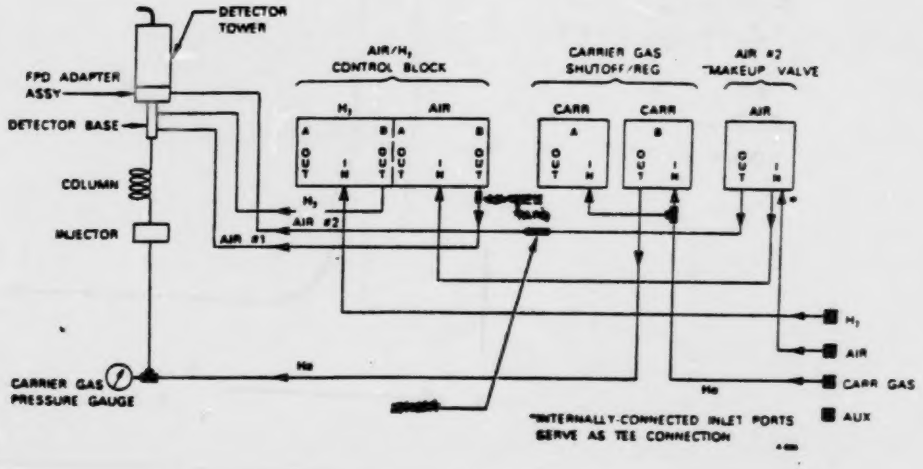

DETECTOR TOWER
FPD ADAPTER ASSY
DETECTOR BASE
COLUMN
INJECTOR
CARRIER GAS PRESSURE GAUGE
AIR/H₂ CONTROL BLOCK
H₂
AIR
A
B
OUT
IN
CARRIER GAS SHUTOFF/REG
CARR
AIR #2 MAKEUP VALVE
H₂
AIR #2
AIR #1
He
H₂
AIR
CARR GAS
AUX
*INTERNALLY-CONNECTED INLET PORTS SERVE AS TEE CONNECTION

Figure 1. Example of a "Status of Instrumentation" Entry.

Reproduced from
best available copy.

$\underline{n}$-tridecane in MeCl$_2$ (15.1 ng/μL)

see next page for column identification

$t'_R = $ ~~#~~ $12.3 - \left(\frac{52.5}{60}\right)2$

$=$ ~~11.4 cm~~ 10.6

GC Conditions

140°C isothermal
helium = carrier gas
52.5 sec sweep time,
8 psi
split ration 15 : 1
column flow = 1.38 mL/min
FID temp : 260°C
inj. temp = 240°C
Split injection
0.2 μL
1024×10^{-12}

theoretical plates

$$n = 5.545 \left(\frac{t_R}{W_h}\right)^2$$

where t_R = retention time of solute

and W_h = width at 1/2 height

$$n = 5.545\left(\frac{123}{1.55}\right)^2 = 34,917$$

Effective theoretical plates

$$N_{eff} = 5.545\left(\frac{t'_R}{W_h}\right)^2$$

where t'_R = adjusted retention time

Sweep time = 52.5 sec

Chart speed = 2 cm/min

$$N_{eff} = 5.545\left(\frac{\overset{106}{\cancel{114}}}{1.55}\right)^2$$

= ~~29,995~~
25,933

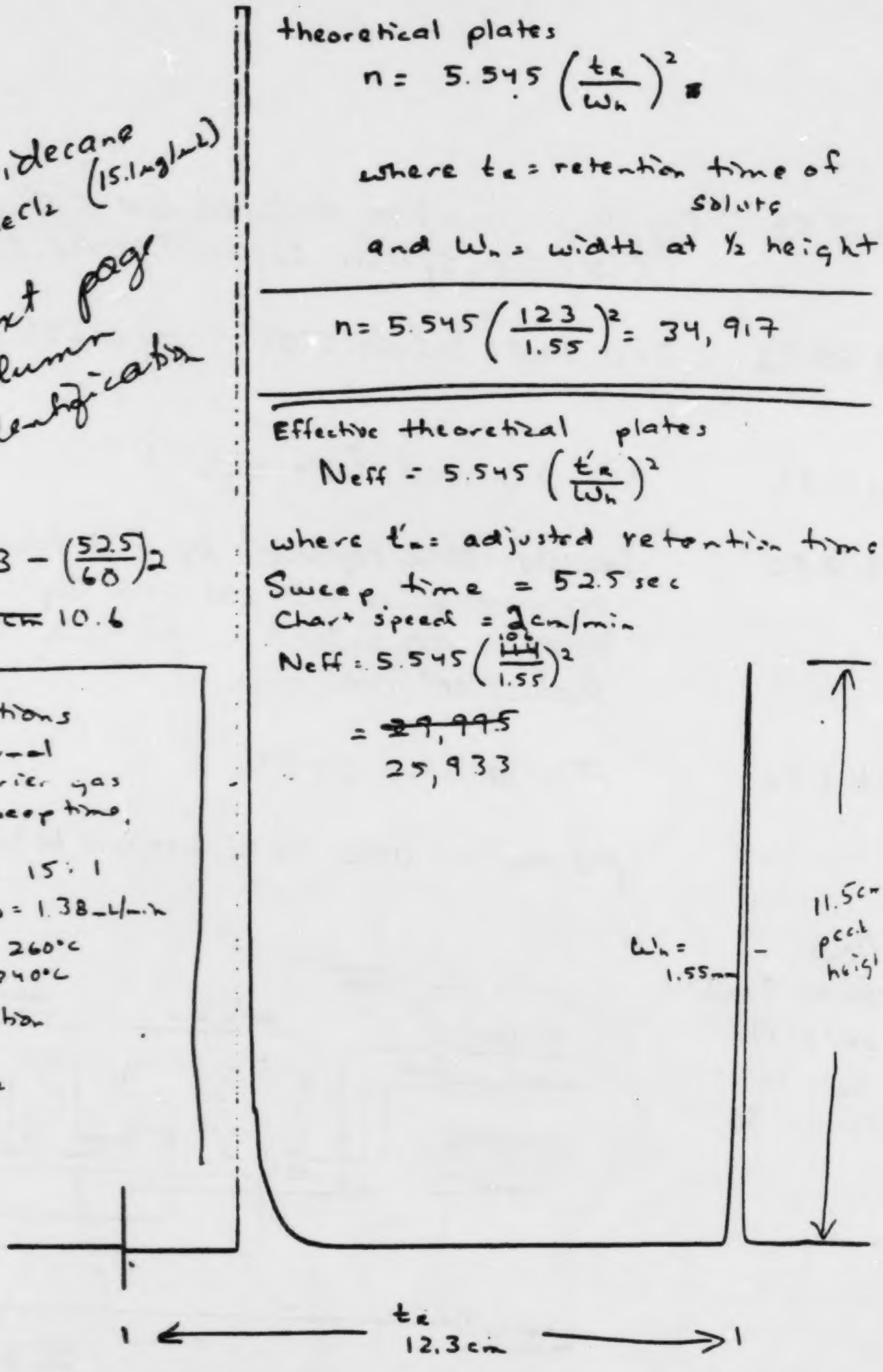
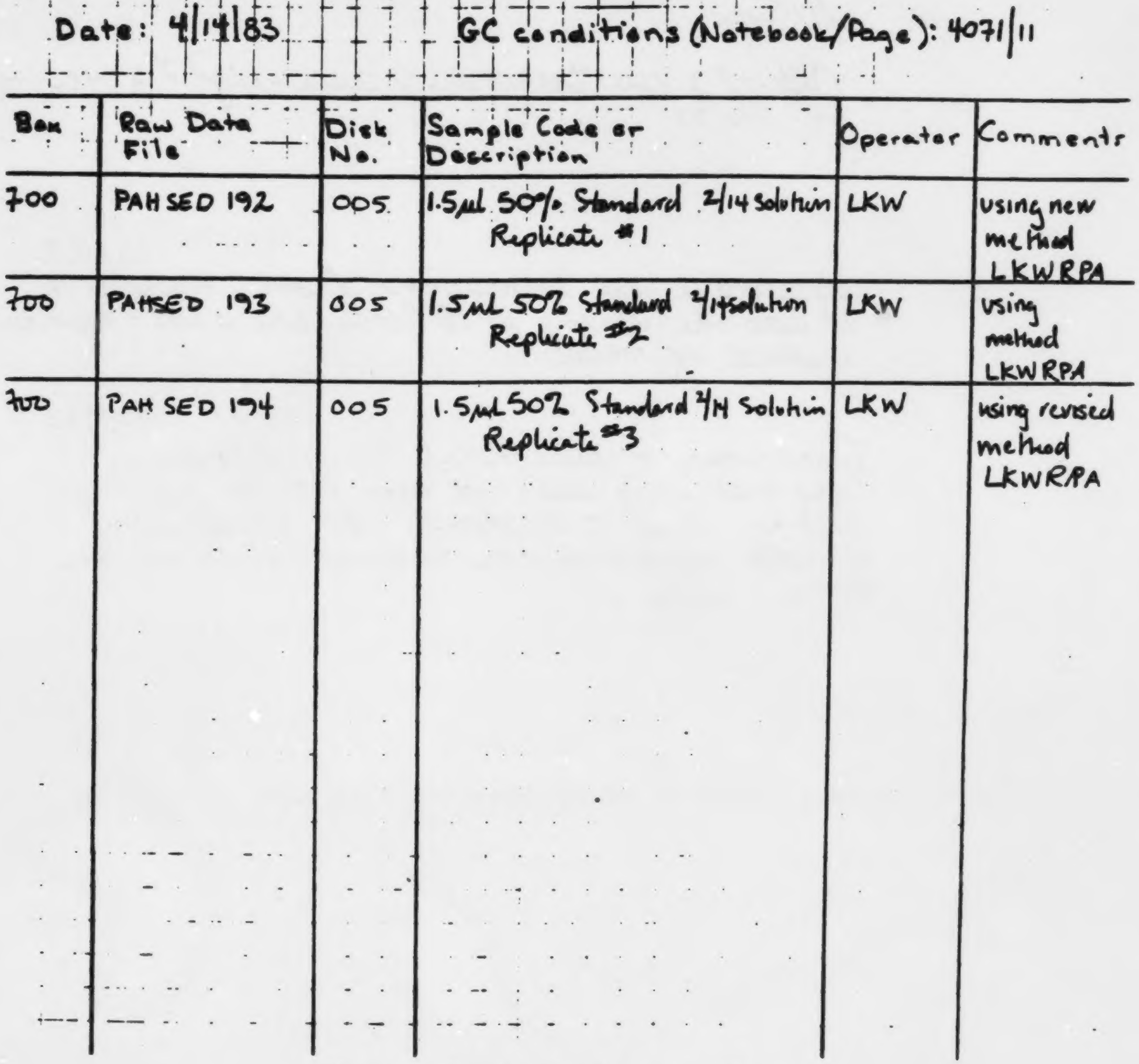


Figure 2. Example of a "Daily Log" Entry.



Date: 4/14/83 GC conditions (Notebook/Page): 4071/11

Box	Raw Data File	Disk No.	Sample Code or Description	Operator	Comments
700	PAH SED 192	005	1.5µl 50% Standard 2/14 solution Replicate #1	LKW	using new method LKWRPA
700	PAHSED 193	005	1.5µl 50% Standard 2/14 solution Replicate #2	LKW	using method LKWRPA
700	PAH SED 194	005	1.5µl 50% Standard 2/14 Solution Replicate #3	LKW	using revised method LKWRPA

Figure 3. Example of a "Sample Log" Entry.

8/30/83

Problem - Pressure meter on M6000A reads >1000 PSI when pump is off. Cannot be zeroed. Tested the transducer according to P. 2-47 of the M6000A Tests & Calibrations - Maintenance Manual & determined that the transducer was defective.

Ordered a new transducer assembly #25480 - on 8/31/83.

9/13/83

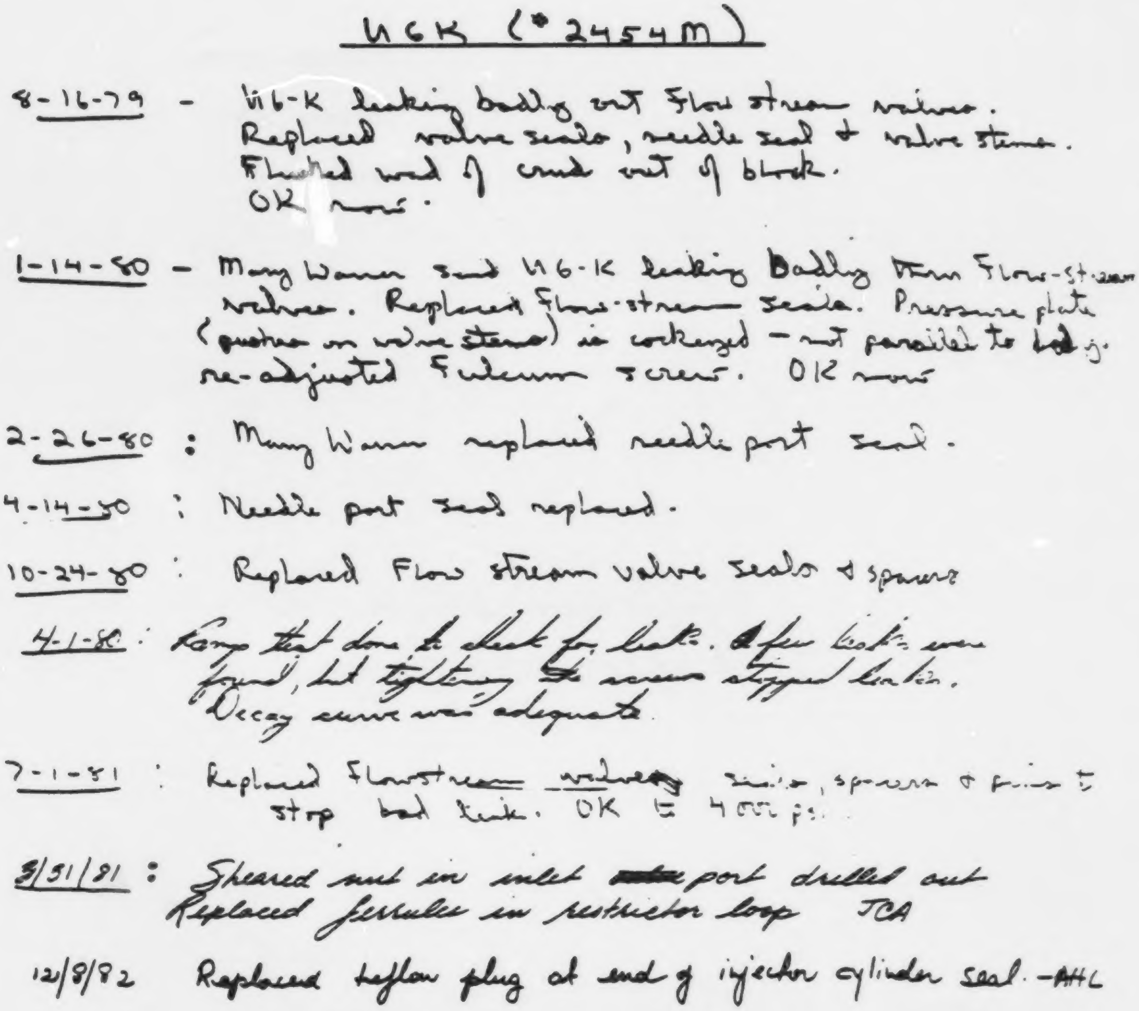
Received a new transducer from Waters & it was the wrong part. (Looks like a WISP transdu instead of M6000A).

9/15/83

Transducer — new model — Steve Parker (regional rep) said it was OK to use. Put in new transducer. It checks out O.K. Zeroed the transducer & compared it to the other pump.

Figure 4. Example of "Instrument Maintenance Log" Entry.

Reproduced from best available copy.



UGK (#2454M)

8-16-79 - U6-K leaking badly out flow stream valves. Replaced valve seals, needle seal & valve stems. Flushed wad of crud out of block. OK now.

1-14-80 - Mary Warren said U6-K leaking badly thru flow-stream valves. Replaced flow-stream seals. Pressure plate (pushes on valve stems) is cockeyed - not parallel to body. re-adjusted fulcrum screw. OK now

2-26-80 : Mary Warren replaced needle port seal.

4-14-80 : Needle port seal replaced.

10-24-80 : Replaced flow stream valve seals & spacers

4-1-80 : Ramp test done to check for leaks. A few leaks were found, but tightening the screws stopped leaks. Decay curve was adequate.

7-1-81 : Replaced flowstream valves seals, spacers & [illegible] to stop bad leak. OK to 4000 psi.

3/31/81 : Sheared nut in inlet [illegible] port drilled out Replaced ferrules in restrictor loop JCA

12/8/82 Replaced teflon plug at end of injector cylinder seal. -AHL

Figure 5. Example of an "Instrument Maintenance Log" Entry.

ACS/SOP-150-001

October 1983

Revision 0

Standard Operating Procedure for Cleaning Glassware/Plasticware

Author:

Robert W. Handy 10/4/83
Robert W. Handy Date
ACS QA Officer

Approved:

E. Pellizzari 10/4/83
E. D. Pellizzari Date
ACS Vice-President

Standard Operating Procedure for Cleaning Glassware/Plasticware

RTI/ACS-SOP-150-001

1.0 INTRODUCTION

Room 170 in Camille Dreyfus Laboratory has been equipped with two triple basin sinks, each supplied with a deionized water line, and two industrial ovens capable of maintaining temperature of 1000°F. ACS laboratory personnel use this facility to clean their dirty glassware and plasticware. The cleaning operation is carried out by an RTI employee assigned to this task.

2.0 SUMMARY OF PROCEDURE

Although several different cleaning procedures have been used in special situations, a standard method has evolved which is used most of the time. The following glassware cleaning procedure consists of two basic parts: soaking the glassware/plasticware in a detergent solution and heating (decontaminating) the glassware in a 900°F oven.

3.0 STANDARD GLASSWARE CLEANING PROCEDURE

3.1 Prepare solution for soaking glassware by mixing 1 cup of Amway detergent with hot water in one of the sink basins.

3.2 Carefully place the glassware from only one laboratory cart in a basin and soak for a minimum of 1 hour. Soak dirty glassware on other carts in separate basins.

NOTE: Expensive or custom-made glassware may be protected by placing item(s) in a basket, beaker or other container during the soaking period.

NOTE: Glassware received during late afternoon may be soaked overnight (or over the weekend).

3.3 Remove any exterior markings and labels and residues with a brush, an abrasive, or other appropriate means.

3.4 Thoroughly rinse the glassware with cold tap water.

NOTE: The criterion for sufficient rinsing is the absence of any soapy water on the glassware. However, there are certain techniques which have proved successful and are described below.

3.4.1 Vessels which hold liquids such as Erlenmeyer or volumetric flasks or narrow-mouth bottles: Fill twice to capacity and empty and then fill to approximately two-thirds capacity and empty while shaking vessel.

3.4.2 Vessels which hold liquids such as beakers, graduates, or wide-mouth bottles: Fill three times to capacity and empty.

3.4.3 Other equipment such as funnels, spatulas, or watchglasses: Rinse while rubbing surface with hands wearing rubber gloves.

3.5 Rinse the glassware with deionized water.

NOTE: This rinsing operation is not as intensive as the cold tap water rinse. Volume of deionized water used during rinse is approximately 1/3 that used of cold tap water.

NOTE: Before it is used in the laboratory, new glassware is often processed by rinsing with deionized water followed by oven heating (Sections 3.8).

3.6 Place clean paper toweling (Crown, singlefold, 9 3/4" x 10 1/4") in the laboratory cart and place rinsed glassware on this toweling to dry.

NOTE: This is the toweling on which the glassware will be placed when removed from the oven (see Section 3.11).

NOTE: Certain pieces of glassware require a longer drying period than others (e.g., beaker vs. volumetric flask). This may be partially overcome by periodically draining any residual water from the vessel on the toweling. This drying operation usually takes 2-3 hours.

3.7 Discharge the soapy water used for soaking down the drain. A fresh soapy water is prepared for soaking glassware from other carts.

3.8 Place glassware in a high temperature oven, close both doors, and turn on heat (maximum temperature, 900° ± 25°F).

NOTE: Both ovens are programmed to heat to 900° ± 25°F, to maintain this temperature, and to shut off two hours after heating cycle began. Thus, the oven temperature is not at maximum during the entire 2 hour period since some of that time is required to heat oven to 900°F.

CAUTION

Do not put any plasticware in the oven. The high temperature will damage the item and contaminate the oven.

3.9 After the 2 hour heating period, open the outer oven doors and allow the oven to cool in this manner for 30 minutes.

3.10 Carefully open the inner oven door and continue to cool glassware for an additional 30 minutes.

NOTE: Both cooling periods may be omitted if the heating cycle is started in late afternoon. Operating in this manner will cause the oven to shut off two hours later and slowly cool overnight (or over the weekend) with the oven doors shut.

CAUTION

Rapid cooling of the oven will increase the likelihood of glassware cracking from extreme temperature changes. Wear asbestos (not rubber) gloves for all oven operations.

3.11 Remove the glassware from the oven and place on paper toweling in the laboratory cart.

NOTE: The toweling was used for the drying operation (see Section 3.6). It has not been customary to cover the clean glassware with anything.

CAUTION

To prevent damage to the glassware, do not remove glassware until the oven has completely cooled.

3.12 Return the glassware to the correct laboratory.

4.0 VARIANCE TO STANDARD CLEANING PROCEDURE

Requests for the use of special cleaning protocols, not covered in the standard method, must be accompanied by a written SOP in which the requested protocol is clearly described. Before implementing the proposed procedure, the SOP must pass through the usual approval chain which consists of the ACS QA Officer and Vice President.

5.0 RULES FOR LABORATORY PERSONNEL

Before sending glassware/plasticware for cleaning, laboratory personnel will perform the following operations:

5.1 Clearly label all laboratory carts used for transporting glassware to and from Room 170. Include lab room number and name of staff member to whom glassware should be returned.

5.2 Place small pieces of dirty glassware on toweling or in a container to prevent breakage on floor.

5.3 Remove all residues from glassware.

5.4 Remove all labels and black markings from glassware.

5.5 Rinse all glassware and containers to remove solvents.

5.6 Writing on frosted sections of glassware should be done in pencil and erased before submitting to washing.

5.7 When clean glassware is returned to the laboratory, empty the cart by placing the glassware in designated storage areas. Dirty glassware should not be put in a cart containing clean glassware.

5.8 Do not place cracked or broken glassware on the cart for washing. Such items will be removed and not processed.

RTI/ACS-SOP-160-001

January 1984

Revision 0

Standard Operation Procedure for the Calibration of a Mettler PL1200 Balance

Author:

Doris J Smith 1/20/84
Doris J. Smith
Date

Approved:

RW Handy 1/20/84
R. W. Handy
ACS Quality Assurance
Date

E Pellizzari 1/20/84
E. D. Pellizzari
ACS Vice-President
Date

Standard Operation Procedure for
the Calibration of a Mettler PL1200 Balance
RTI/ACS-SOP-160-001

1.0 INSTRUMENTATION

The Mettler PL 1200 has a weight range of 0 to 1200 g with a readability of 0.01 g. It has an additional taring range of 100 g so that the maximum load is 1300 g.

2.0 GENERAL INSTRUCTION MANUAL

A copy of the instruction manual is filed in Lab 180. In addition, instructions for operation of the balance are attached to the balance (a copy is included in this SOP with a general view and top view of the instrument).

3.0 CALIBRATION

Calibration must be checked each time the balance is moved, and periodically (every month, minimum) depending upon usage and project protocol.

3.1 Place a standard 100 g weight (±0.2 g) on pan and set built-in weight control knob ("14" on general view, Figure 1) to "1".
NOTE: The standard weight must be Class S-1 or equivalent.

3.2 Adjust to exact zero with zero-adjustment disk ("20" on general view, Figure 1).

3.3 Return weight-control knob to "0" and read results on the optical scale. The optical scale must read 100 g exactly.

3.4 If the reading is less than 100 g, remove housing cover and raise weight (25 on top view, Figure 2).

3.5 If reading is more than 100 g, remove housing cover and lower weight (25 on top view, Figure 2).

3.6 Repeat calibration steps until optical scale reads 100 g exactly.

4.0 DOCUMENTATION

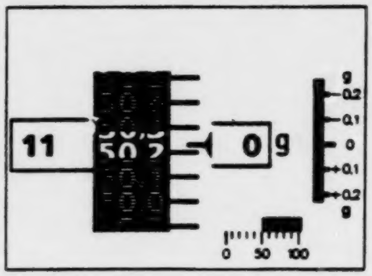
4.1 A hard-bound book must be maintained for balance records.

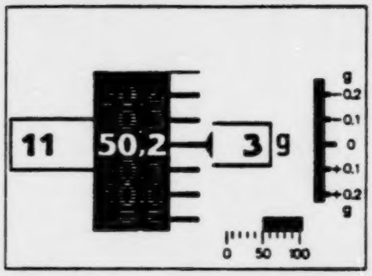
4.2 All calibration information must be kept in the hard-bound book, including:

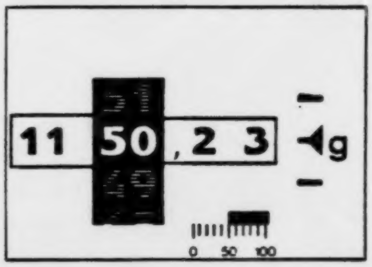
- 4.2.1 Date of calibration,
- 4.2.2 Name of person performing the calibration,
- 4.2.3 Results of the procedure.

Directions for use

Switch light ⑩ on.
Balance should be level. Adjust if necessary.
Turn tare knob ⑬ in direction of arrow to stop position.
Set digital result to "0" with digital-counter knob ⑮.
Set built-in weight result ㉗ to "0" with built-in weight-control knob ⑭.
Adjust zero line of optical scale exactly to index mark of ground-glass plate with zero-adjustment disk ⑳.







1. To weigh a sample

- Place sample to be weighed on pan.
- If plus signs appear on the optical scale, turn the built-in weight-control knob ⑭ until numbers appear on the optical scale.
- Read the result.

Analog readout

The final digit, ten milligrams, is estimated by determining the position of the pointer between scale divisions.

Sample weight: 1150,22 g

Digital readout

Turn the digital-counter knob ⑮ until the next lower scale division lines up with the pointer.

Sample weight: 1150,23 g

2. To tare a sample

Samples weighing up to 100 g may be tared by turning the tare knob ⑬ continuously back to zero.
If the tare is more than 100 g, the built-in weights have to be used.
When using the built-in weights, the corresponding value has to be subtracted off the weighing result.
The red pointer or the red " T " resp. indicates the range used in the tare indicator ㉛ .

Instructions for Using a Mettler PL1200 Balance

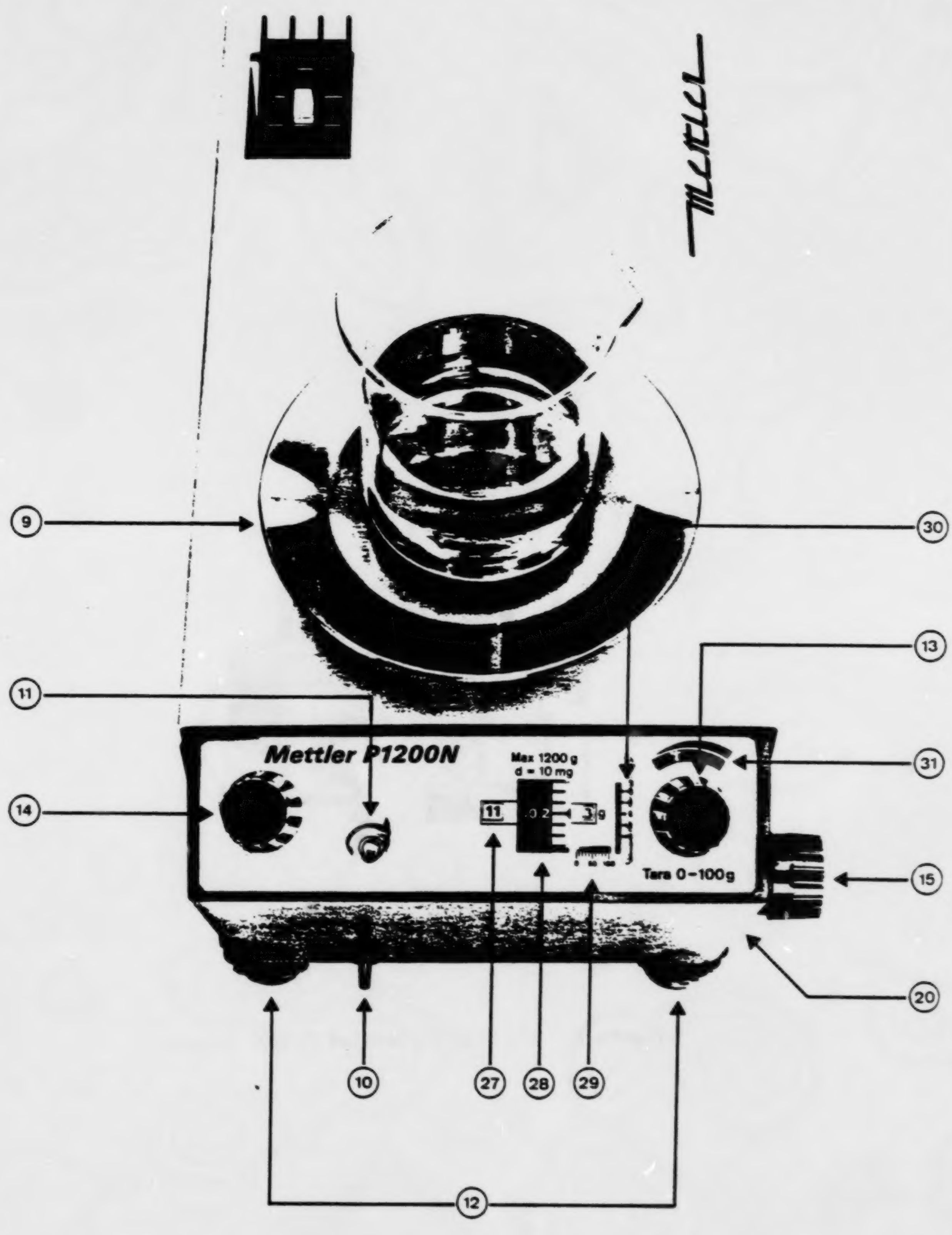

METTLER
Mettler P1200N
Max 1200 g
d = 10 mg
11
.0 2
3 g
Tara 0–100 g
9
30
13
11
31
14
15
20
10
27
28
29
12

Figure 1. General view of the Mettler PL1200

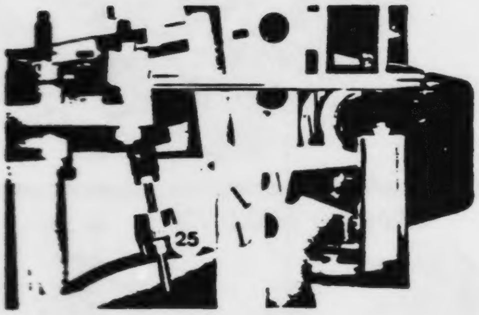


Figure 2. Top View of Mettler PL1200 Balance

RTI/ACS-SOP-160-002

January 1984

Revision 0

Standard Operating Procedure for the Calibration of a Mettler M5 Analytical Balance

Author:

Doris J Smith 1/20/84
Doris J. Smith
Date

Approved:

RW Handy 1/20/84
R. W. Handy
ACS QA Officer
Date

E Pellizzari 1/20/84
E. D. Pellizzari
ACS Vice-President
Date

Standard Operating Procedure for the
Calibration of a Mettler M5 Analytical Balance
RTI/ACS-SOP-160-002

1.0 INSTRUMENTATION

The Mettler M5SA is a micro analytical balance with a range of 0-20 g and a readability of 2 μg.

2.0 GENERAL INSTRUCTION MANUAL

A copy of the instruction manual is kept in room 194. Instructions for operation of the balance are attached to the balance (a copy is included in this SOP).

3.0 CALIBRATION

Calibration must be checked whenever balance is moved and a minimum of twice a year depending upon usage and project protocol.

NOTE: The standard weight must be Class S-1 or equivalent.

3.1 Load pan with a standard 10 mg weight (± 0.3 mg) and adjust knob D to 0.01 g (see Figure 1).

3.2 Release the balance fully and set zero point accurately.

3.3 With the balance released, carefully turn knob D back to 0.

3.4 If the resultant deflection is less than 100 graduations, arrest balance, and turn adjusting weight 5 counter-clockwise. Repeat operations 3.1 to 3.3 until a deflection of 100 graduations is reached.

3.5 If the deflection is greater than 100 graduations, arrest balance, and turn adjusting weight 5 clockwise. Repeat operations 3.1 to 3.3 until a deflection of 100 graduations is reached.

WARNING

After touching beam or adjusting screw, wait about 15 minutes with further measurements to allow temperature of beam to equalize.

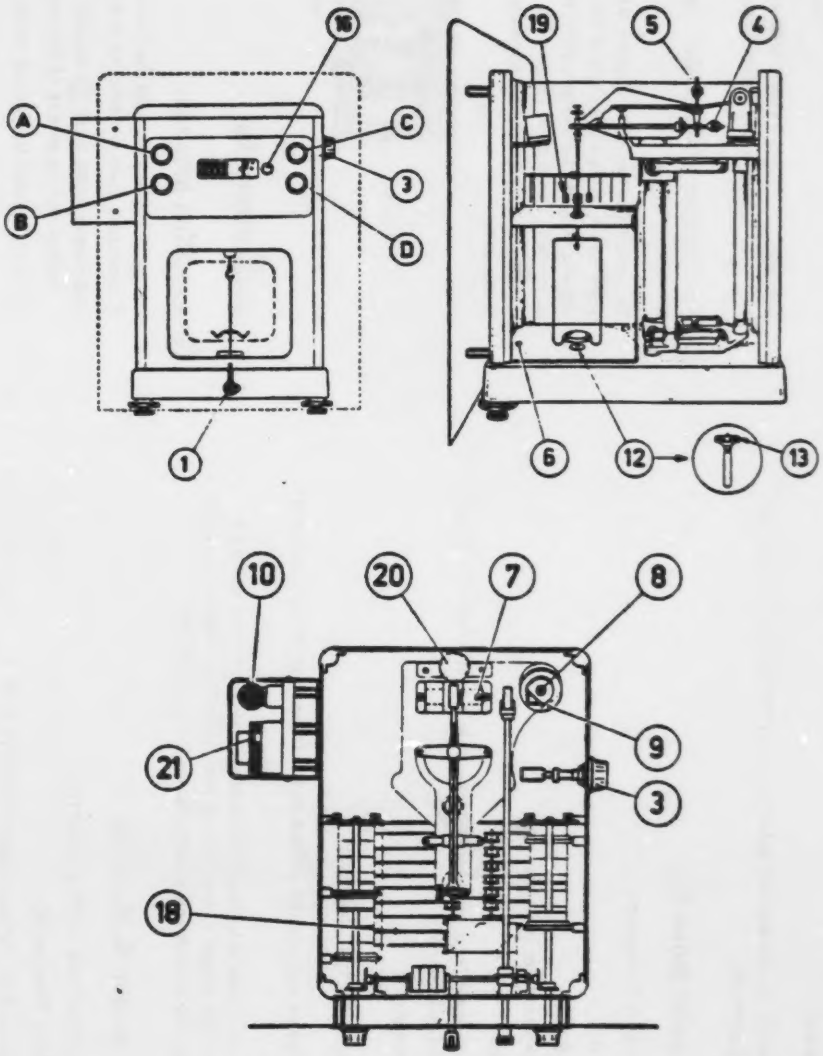

16
A
C
3
B
D
1
19
5
4
6
12
13
10
20
7
8
9
21
3
18

Figure 1. Diagram of Mettler M5 Balance.

INSTRUCTIONS FOR USING A METTLER M5 BALANCE

Weighing

Weighing requires the following operations:

❶ Checking the level

Check the spirit level ⑥ to ensure that balance is properly levelled. If necessary, relevel balance (see Levelling).

❷ Checking zero point (knob ③)

a) Unload pan and clean it if necessary

b) Close side window

c) Set all weights to 0

d) Set micrometer scale to 0 with knob ⑯

e) Release balance fully

f) When the projected scale is at rest, bring 0-line of scale with knob (3) exactly into centre of forked pointer

g) Arrest balance (Position I)

❸ Laying on objects to be weighed

The object to be weighed may only be placed on the balance in the arrested state (Position I).

Whenever possible use a pair of tweezers to avoid humidity and heat being transmitted by the hands to the object to be weighed and inside the weighing chamber. After introducing object to be weighed, close side window immediately.

❹ Weight setting (knobs A, B, C, D)

a) Arrest balance and turn knob A (10 g) to the right

b) Semi-release balance (Position II)

c) If scale moves upward, arrest balance and turn back knob A

d) If scale moves downward, turn knob B clockwise until scale moves upward. Turn back knob B by one place

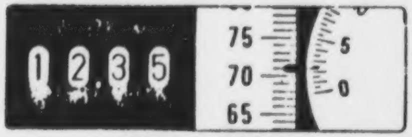
e) Repeat same operation with the remaining knobs for weight setting

f) Arrest balance after having set the weights. (In this position, the pan brake becomes operative)

g) Release balance fully and allow it to come to rest

h) On M5, reading of last two decimal places is done with the aid of the micrometer as follows:
Turn micrometer knob anti-clockwise until next lower graduation lies exactly in center of forked pointer

Fig. 1



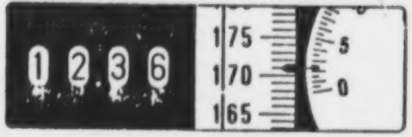
Weight 12,357123

Caution: To avoid unnecessary heating, do not keep fingers on knob longer than necessary. Never follow up movement of scale with micrometer.

❺ Facilitating differential weighing operations in the optical range

So as to make the most of the ± 0.002 mg accuracy of the optical range, the optical scale has a range of 20 mg and 200 graduations respectively. Thus, without operating the weight mechanism, differential weighing operations of at least 10 mg are possible in the optical range. The scale range between 10 and 20 mg has a black line. When this black line appears, one unit must be added to the last number of the counter.

Fig. 2



Weight = 12,377123

Adjustments

❶ Levelling balance

Before doing any work on the balance, check whether it is properly levelled.
If necessary, level the balance as follows:

a) Observe spirit level ⑥ through front glass and adjust spirit level in the longitudinal axis by means of the two front foot screws

b) Using the rear foot screw, bring spirit level exactly into circle

❷ Zero point

With adjusting knob ③, the 0-line can be displaced by ± 3 graduations. If the deviation present is greater, i. e. no longer capable of being corrected with knob ③, then proceed as follows:

a) Level balance accurately. (If balance was badly out of level, try once more to adjust zero point with knob ③

b) Remove cover

c) With balance arrested, adjust fine taring weight ④. Turning fine taring weight clockwise raises the 0-line. Turning it counter-clockwise lowers the 0-line

RTI/ACS-SOP-160-003

January 1984

Revision 0

Standard Operating Procedure for the Calibration of a Torbal EA-1 Balance

Author:

Doris J. Smith 1/20/84
Doris J. Smith Date

Approved:

RW Handy 1/20/84
R. W. Handy Date
ACS QA Officer

E Pellizzari 1/20/84
E. D. Pellizzari Date
ACS Vice-President

Standard Operating Procedure for
the Calibration of a Torbal EA-1 Balance
RTI/ACS-SOP-160-003

1.0 INSTRUMENTATION

The torbal EA-1 is a torsion balance with a range of 0-160 g and a 0.1 mg readability.

2.0 GENERAL INSTRUCTION MANUAL

A copy of the instruction manual is kept in Room 194. Instructions for operation of the balance are kept proximate to the balance (a copy is attached is this SOP).

3.0 CALIBRATION

Even though the "Range" remains constant for extended periods, it is good weighing practice to check it a minimum of twice a year and adjust if necessary.

3.1 Turn balance on and note that pilot light is on. Allow 15 minutes for balance to warm up.

3.2 Set all weight control knobs (5) to zero and rotate the counter control knob (8) to 000 on the counter (9)(see Figure 1). Bring the null indicator needle to the center line or null by operating the coarse tare and zero adjustment knobs.

3.3 Place a standard 100 mg class S-1 weight (or equivalent) on the balance pan, close the balance door and operate the counter control knob (8) to a setting of 999 (see Figure 1). The null indicators needle should come to rest at center or within a needle width to the left of center.

3.4 If, in checking the counter range, the counter setting of 999 causes the pointer to be more than a pointer width to the left or at any point to the right of null, it will be necessary to adjust the range control potentiometer. (An access hole is provided in the lower portion of the rear shroud of the balance.) Use a screwdriver and rotate the slotted shaft slowly until the pointer returns to the null position.

3.5 Reset the counter (9) to 000, remove the 100 mg weight or advance the right weight control knob (5c) one number, and use the zero adjust knob to place the needle at null. Repeat the test and readjust if required.

4.0 DOCUMENTATION

4.1 A hard-bound book must be maintained for balance records.

4.2 All calibration information must be kept in the hard-bound book, including:

- 4.2.1 Date of calibration,
- 4.2.2 Name of person performing the calibration,
- 4.2.3 Results of the procedure.

INSTRUCTIONS FOR USING A TORBAL EA-1 BALANCE

Weighing Procedure for Balance

1. Level the balance by manipulating the two front leveling feet.
2. Switch on power at toggle switch on lower right side of balance; the indicator light on the back of the weighing chamber should come on. Allow the balance to warm up for 15 minutes. Leave it turned on all day.
3. Turn all weight control knobs and counter control knob to zero.
4. Use coarse tare control on upper left side of balance and zero adjust knob on lower right side of balance to bring the null indicator to zero.
5. Open door and place sample on balance pan. Close door.
6. Starting with the left weight control knob, increase the reading by turning the knob clockwise until the null indicator needle swings from "INCREASE" to "DECREASE". Reduce the digital reading by one and proceed to the center knob then the right knob, repeating the above procedure. Rotate the counter control knob to bring the null indicator to zero.
7. Leave the balance with the null indicator at zero (as in Step 4) when you are finished.

Use of Tare Devices

When taring an object set all number wheels and counter at zero.

The tare indicator located on the upper left front of the balance is not calibrated. The position of the indicator needle shows the approximate amount of tare in use.

The total amount of tare that can be obtained by rotating the tare control is approximately 8 grams.

To tare objects whose weight is between 8 and 20 grams, replace the stainless steel pan with the aluminum pan. Place object on the aluminum pan. Add rectangular tare weights one at a time until the null indicator needle moves from "DECREASE" to "INCREASE". Adjust the zero of the balance using the tare ajust knob and th zero adjust. .

To tare objects whose weight is between 20 and 48 grams, remove the weighing pan, place object on the "underpan" and add the rectangular tare weights, one at a time, until the null indicator needle moves from "DECREASE" to "INCREASE". Adjust the zero of the balance using the tare adjust knob and the zero adjust.

The aluminum pan and tare weights when not in use should be on the hook on the rear panel of weighing chamber. The numbers on the tare weights are approximate weight values.

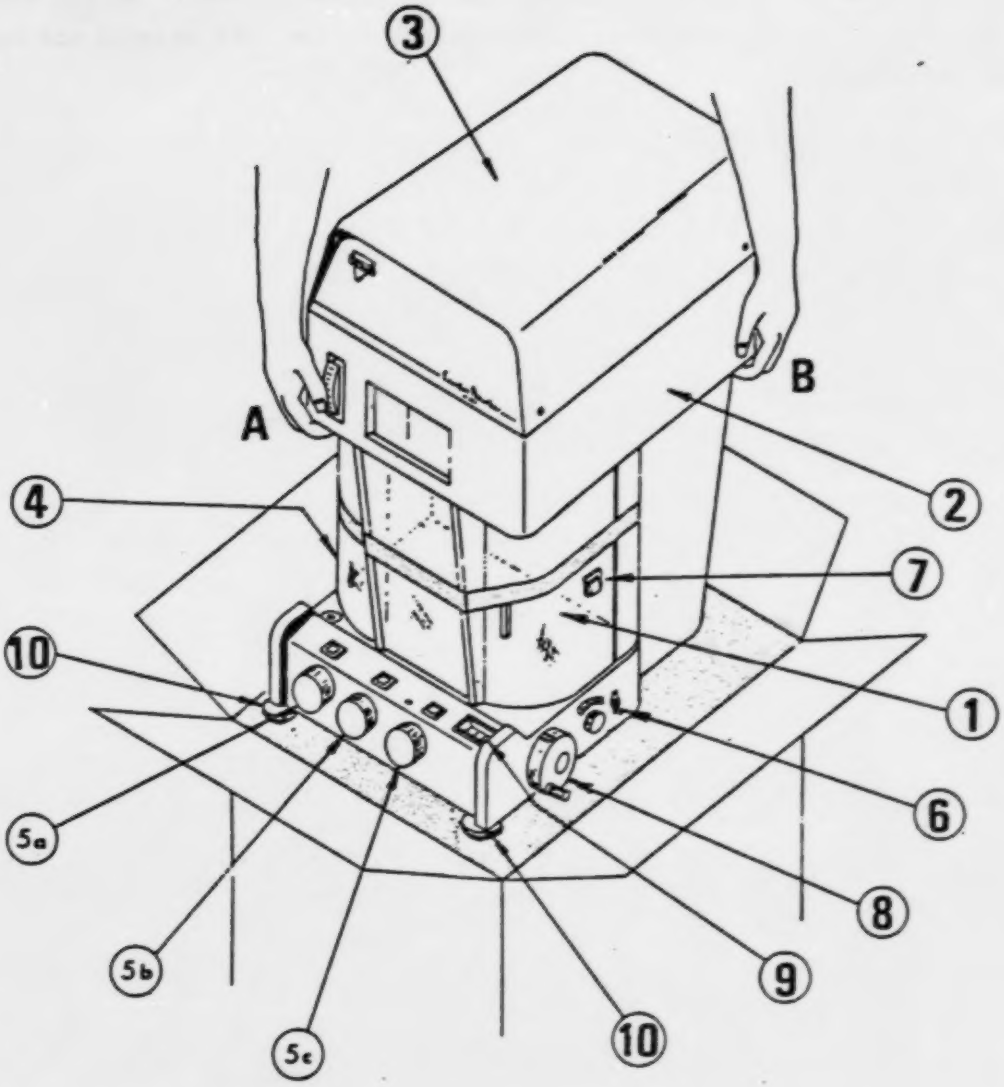

③
A
B
②
④
⑦
⑩
①
⑥
5a
⑧
5b
⑨
5c
⑩

Figure 1. Diagram of Torbal EA-1 Balance

RTI/ACS-SOP-160-004

January 1984

Revision 0

Standard Operating Procedure for the Calibration of a Mettler PL200 Balance

Author:

Doris J Smith 1·20·84
Doris J. Smith Date

Approved:

RWHandy 1/20/84
R. W. Handy Date
ACS QA Officer

E Pellizzari 1/20/84
E. D. Pellizzari Date
ACS Vice-President

Mini-Standard Operating Procedure for the
Calibration of Mettler PL200 Balance
RTI/ACS-SOP-160-004

1.0 INSTRUMENTATION

The Mettler PL200 has a weight range of 0 to 220 g with a readability of 0.001 g.

2.0 GENERAL INSTRUCTION MANUAL

The instruction manual is kept in Lab 105 proximate to the balance. Instructions for operation of the balance are attached to the balance (a copy is included in this SOP).

3.0 CALIBRATION

Calibration should be checked each time the balance is moved and a minimum of once a month, depending upon usage and project protocol.

3.1 Switch on balance by pressing down control bar.

WARNING

> Prior to calibration, the balance must remain switched on for about 20 minutes.

3.2 Fine-Calibration by Means of the Calibration Screw

Standard weights (2 x 100 g ± 1 mg) class S-1 or equivalent are needed to calibrate the balance.

- 3.2.1 Tare the balance (press down control bar), readout indicates 000.000 g.
- 3.2.2 Place the 2 x 100 g standard weights on the weighing pan. If the readout does not indicate 200.000 g, the balance must be calibrated in the following manner:
- 3.2.3 Insert screwdriver in calibration hold. Depending on the deviation, turn screwdriver in appropriate direction. One turn changes the calibration by about 0.05 g (50 digits).
  - -In case of positive deviation (readout indicates more than 200 g), turn screw counterclockwise.
  - -In case of negative deviation (readout indicates less than 200 g), turn screw clockwise.

-After every adjustment with the calibration screw, press control bar first, then place calibration weight on pan. Read indicated value and, if necessary, calibrate again.

NOTE: The balance is correctly calibrated when the readout indicates 000.000 g (without adjusting the calibration screw) and + 200.000 g with the calibration weights in the pan.

3.3 Coarse-calibration by Means of a Coding Plug

If the balance cannot be calibrated with the calibration screw (insufficient calibration range), proceed as follows:

3.2.1 Screw in calibration screw all the way to stop position, then screw out by 10 turns (center position).

3.2.2 Place 200 g calibration weight on pan and calculate deviation from ideal value.

3.2.3 Disconnect power cable and open housing cover.

3.2.4 Adjust coding plug according to deviation from ideal value. If the plug is plugged in lower, the calibration becomes smaller, if it is plugged in higher, the calibration gets larger. Each step changes the calibration by 0.2%, i.e. for the PL200 the change is 0.4 g or 400 digit.

3.2.5 Install housing cover, plug in power cable and calibrate balance with calibration screw.

3.4 If the balance cannot be calibrated with the calibration screw or the coding plug, the calibration weight is wrong or the measuring cell of the balance is defective. In the latter case inform nearest Mettler service facility.

4.0 DOCUMENTATION

4.1 A hard-bound book must be maintained for balance records.

4.2 All calibration information must be kept in the hard-bound book, including:

4.2.1 Date of calibration,

4.2.2 Name of person performing the calibration,

4.2.3 Results of the procedure.

INSTRUCTIONS FOR USING A METTLER PL200 BALANCE

Switching the balance on and on

-Switching on the balance:press control bar:the readout indicates 888.888 g (:888.88 g) and then 000.000 g (000.00 g). When the 888.888 g (1888.88 g) light up, it is possible to check the proper functioning of the 7-segment display including the sign.

-Switching off the balance:lift control bar.

NOTE: In case of a power failure, the balance switches off automatically. It must then be switched on by pressing the control bar.

Weight determination

-Press down control bar:readout indicates 000.000 g (000.00 g).

-Place sample on pan; - Read weight of sample on readout.

-If the maximum load has been exceeded, the readout will be blanked out, except for the decimal point.

Taring and weighing-in

-Place container to be tared on weighing pan.

-Press down control bar. Readout indicates 000.000 g (000.00 g).

-While adding material, observe only that part of the readout in which the numbers change slowly.

-Shortly before reaching the desired weight, observe the entire readout.

-If maximum load is exceeded, readout will be blanked out except for the decimal point.

-The weighing capacity corresponds to the weighing range minus the weight of the tared object.

Checkweighing

-Place reference weight on weighing pan.

-Press down control bar. Readout indicates 000.000 g (000.00 g).

-Remove reference weight. Value of reference weight is now indicated with negative sign on readout.

-Place object to be checked on weighing pan.

-Any negative deviation from reference weight is now indicated by the minus sign lighting up in front of the weight indication; positive deviation is shown by the lighting up of the plus sign.

Example: Reference weight 100 g Object to be checked a) 93 g or b) 104 g

a) Deviation (readout): -100 + 93 = -7 g

b) Deviation (readout): -100 + 104 = +4 g

RTI/ACS-SOP-320-001
December 1983
Revision 0

Standard Operating Procedure for Tenax Cleanup and Preparation of Tenax Cartridges for Use in the Collection of Organic Compounds

Author:

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Approved:

C M Sparacino 3/15/84
Laboratory Manager, RTI/ACS Date

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Standard Operating Procedure for Tenax Cleanup and Preparation of Tenax Cartridges for Use in the Collection of Organic Compounds

RTI/ACS-SOP-320-001

1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is two-fold: (1) it describes the methodology for cleaning Tenax and (2) it describes step-by-step the preparation and purification of the Tenax cartridges.

2.0 SUMMARY OF THE METHOD

All Tenax, whether new or recycled, must be purified before it is used for sample collection of organic compounds. The following routine shall be followed when Tenax is cleaned and packed into cartridges: (1) selection of the Tenax to be used; (2) solvent extraction; (3) drying the Tenax; (4) sieving the Tenax; (5) packing the Tenax into glass cartridges; (6) thermally desorbing the Tenax cartridges; (7) ensuring the integrity of the cleaning and desorbing procedure; and (8) packing and storing the cartridges.

3.0 CLEANING THE TENAX

3.1 If new Tenax is to be used follow Steps 3.1.1 to 3.1.3

3.1.1 Assign a unique number to the batch of Tenax to be cleaned.

NOTE: Tenax is subjected to the different steps of the cleaning procedure by batch.

3.1.2 In a bound RTI notebook assigned for Tenax only, begin a section every time a new batch is created.

3.1.3 Keep a record of the Tenax Lot number used.

NOTE: Before a new lot of Tenax is purchased, a small amount is subjected to the cleanup procedure and the background checked for acceptability.

3.2 If the Tenax to be cleaned is recycled Tenax follow Steps 3.2.1 to 3.2.4.

3.2.1 Select Tenax from the same matrix that was previously sampled (i.e., breath, personal air, fixed-site air).

3.2.2 Assign a unique batch number to the quantity to be used.

3.2.3 In the Tenax RTI notebook, begin a section for the new batch.

3.2.4 Enter the complete history of the Tenax (e.g., number of times used, including date(s) of preparation, number of cartridges in the last batch, etc...).

CAUTION

If a Tenax batch was previously known to be dirty, do not mix with another batch.

3.3 Solvent Extraction

3.3.1 Solvents

3.3.1.1 Methanol, B&J distilled in glass

3.3.1.2 n-Pentane, B&J distilled in glass

3.3.2 Apparatus and Materials

3.3.2.1 Extraction thimbles, cellulose (60 mm x 180 mm)

3.3.2.2 Glass wool, unsilanized

3.3.2.3 Soxhlet extractor, large 71/60 joint

3.3.2.4 Flask, round bottom 1000 mL

3.3.2.5 Condenser

3.3.2.6 Tweezers

3.3.2.7 Beaker, 100 mL

3.3.2.8 Variable transformer

3.3.2.9 Heating mantle for 1000 mL flask

NOTE: All glassware must be cleaned by soaking for at least one hour in Amway SA-8 Laundry compound, followed by several rinses with deionized water; and finally, baking for a minimum of four hours at 550-550°C.

3.3.3 Procedure

3.3.3.1 In a hood, set up a sufficient number of Soxhlet extraction units, each with a 1000 mL round bottom flask and a water cooled condenser.

3.3.3.2 Load approximately 50 g of Tenax into each thimble.

3.3.3.3 Cover the Tenax with approximately two centimeters of unsilanized glass wool.

3.3.3.4 Place the thimble in the Soxhlet.

3.3.3.5 Add 600 mL of methanol to the 1000 mL flask.

3.3.3.6 Carefully pour an additional 300 mL of methanol onto the Tenax.

NOTE: The 300 mL of extra methanol are added directly onto the Tenax to ensure sufficient solvent for the extraction process after the initial adsorption of solvent.

3.3.3.7 Turn on the water to the condenser.

3.3.3.8 Turn on the Variac controlled heating mantle.

3.3.3.9 After the first extraction cycle, adjust the temperature with the variable transformer to obtain five cycles per hour.

3.3.3.10 Record in the Tenax notebook the date and time the extraction was started.

3.3.3.11 Begin a Tenax cleanup worksheet (see Example 1) and keep this worksheet in the laboratory next to the hood.

3.3.3.12 Continue the extraction for 48 hours.

3.3.3.13 Check the extraction units twice daily and enter the information on the worksheet.

CAUTION

To avoid solvent losses, ensure that sufficient water is flowing to cool the condensers.

3.3.3.14 After 48 hr cool the system and discard the methanol.

3.3.3.15 With a pair of tweezers carefully pull out the thimble and let it drain in a 100 mL beaker for 10 minutes.

3.3.3.16 Rinse the thimble with 50 mL of clean pentane. Repeat the rinse twice and then return the thimble to the Soxhlet. Discard the pentane.

CAUTION

To avoid contamination do not handle the thimble with your hands.

3.3.3.17 Transfer 700 mL of clean pentane to the flask. Reposition the Soxhlet and heat to reflux.

3.3.3.18 After the first cycle, adjust the temperature to obtain five cycles per hour.

3.3.3.19 Record in the notebook the date and time that the pentane extraction began.

3.3.3.20 Complete the information on the worksheet for this Tenax batch.

3.3.3.21 Continue the extraction for 48 hr.

3.3.3.22 Check the extraction units twice daily and enter the information on the worksheet.

3.3.3.23 After 48 hr of extraction, cool the system to room temperature.

3.3.3.24 Remove the thimble from the Soxhlet with a pair of tweezers.

3.3.3.25 Discard the pentane.

3.4 Drying the Tenax

3.4.1 Apparatus and Materials

3.4.1.1 Desiccator with gas connectors

3.4.1.2 Jar, wide mouth amber

3.4.1.3 Crystallizing dish, Kimax®

3.4.1.4 Vacuum oven equipped with a dry ice trap

3.4.1.5 Aluminum foil

3.4.1.6 Funnel

3.4.2 Procedure

3.4.2.1 Place the beakers containing the thimbles in the desiccator at room temperature under a slow "house" nitrogen flow (i.e., 25 mL/min).

CAUTION

Ensure that the activated charcoal in the carbon trap set up on line is replaced every three months.

3.4.2.2 The following day transfer the contents of two thimbles to a large crystallizing dish.

3.4.2.3 Transfer the rest of the Tenax to a wide mouth jar and label it indicating that has not been dried.

3.4.2.4 Cover the dish loosely with aluminum foil.

3.4.2.5 Set the dish in the vacuum oven.

3.4.2.6 Place dry ice/isopropanol in the vacuum trap.

3.4.2.7 Dry the Tenax overnight at 100°C and 28 inches of water.

3.4.2.8 The following day turn off the heater and allow the oven to reach room temperature before opening the oven.

NOTE: The oven needs approximately 3 hours to cool to room temperature.

3.4.2.9 To open the vacuum oven, first close off the valve leading to the pump.

3.4.2.10 Connect the "house" nitrogen line to the other valve connector on the vacuum oven.

3.4.2.11 Slowly turn on the nitrogen flow with one hand while opening the valve with the other hand.

NOTE: This procedure allows the oven to reach normal pressure under a nitrogen atmosphere.

CAUTION

Ensure that the nitrogen is vented out the oven through an activated charcoal tube.

3.4.2.12 Record every operation on the "Tenax Cleanup Worksheet" and in the notebook.

3.4.2.13 Remove the Tenax from the vacuum oven.

3.4.2.14 Open the valve leading to the pump and then immediately turn the vacuum pump off.

3.4.2.15 Carry the Tenax to the "clean room" and store it protected from the light, in a clean wide mouth jar with Teflon-lined cap.

3.4.2.16 Dry the rest of the Tenax batch following the steps 3.4.2.2 to 3.4.2.15.

4.0 PREPARATION OF TENAX CARTRIDGES

4.1 Sieving

4.1.1 Apparatus and Materials

4.1.1.1 Cotton gloves

4.1.1.2 Sieves, 40 and 60 mesh

4.1.1.3 Glass funnel

4.1.2 Procedure

4.1.2.1 Combine the contents of the jars containing Tenax from the same batch.

4.1.2.2 Sieve the material and collect the contents in the 40/60 mesh range.

4.1.2.3 Return the contents to the jar. Label the jar "sieved" and indicate the date.

4.1.2.4 Record this operation in the notebook.

4.2 Packing

4.2.1 Materials

4.2.1.1 Cotton gloves

4.2.1.2 Glass wool, unsilanized

4.2.1.3 Kimax culture tubes, 15 cm x 2.5 cm O.D.

4.2.1.4 Teflon cap liners, 24 mm new

4.2.1.5 Stainless steel tweezers

4.2.1.6 Screw caps, 24 mm

4.2.1.7 Silicone septa, Teflon-backed

4.2.1.8 One gallon metal paint cans

4.2.1.9 Glass tubes, 10 cm x 1.6 cm O.D.

4.2.2 Preparation of the Culture Tubes

4.2.2.1 Place the Teflon liners in a beaker and sonicate them in methanol for 10 minutes.

4.2.2.2 Rinse the liners with fresh methanol.

4.2.2.3 Repeat Steps 4.2.2.1 and 4.2.2.2 with pentane instead of methanol.

4.2.2.4 Dry the Teflon liners in the vacuum oven for five hours at 100°C and 28 inches of water.

4.2.2.5 Store the liners in a wide mouth jar in the "clean room".

CAUTION

To avoid contamination of the Tenax, always use a pair of tweezers to handle the liners.

4.2.2.6 Follow steps 4.2.2.1 to 4.2.2.5 to clean the silicone septa.

4.2.2.7 Soak the 24 mm screw caps in methanol for 30 minutes.

4.2.2.8 Remove the paper-lined foil from the caps with a spatula.

4.2.2.9 Rinse the caps in clean methanol and dry them in the vacuum oven overnight at 100°C.

4.2.2.10 Wrap the Kimax culture tube with aluminum foil and secure it with clear tape.

4.2.2.11 Place a 2-cm glass wool plug at the bottom of the culture tube.

4.2.2.12 Place a silicone septum in the screw cap. Cover the septum with a cleaned Teflon-liner.

4.2.2.13 Loosely close the culture tube with the screw cap.

4.2.3 Cartridge Packing

4.2.3.1 Carefully inspect the glass tubes before packing. Discard any tube with rough ends or cracks.

4.2.3.2 Set the glass tubes in a test tube rack.

4.2.3.3 Insert a 1-cm glass wool plug into one end of the glass tube and press with a dowel.

4.2.3.4 Transfer 6 cm of Tenax to the glass tube, using a glass funnel.

4.2.3.5 Insert another 1 cm glass wool plug into the other end of the tube (see Figure 1). Lightly compress it with a dowel.

NOTE: A 10 cm glass tube packed with Tenax is referred to as a Tenax cartridge.

4.2.3.6 Store the Tenax cartridges in the prepared culture tubes until desorption.

4.3 Desorption

4.3.1 Equipment and Materials

4.3.1.1 Desorption chambers

4.3.1.2 Helium, (certified 99.995%), with regulator

4.3.1.3 Tweezers

4.3.1.4 Clean paint cans, 1 gallon

4.3.2 Procedure

4.3.2.1 Turn on the desorption units to 270°C.

4.3.2.2 Turn on the helium tank.

4.3.2.3 Place liquid nitrogen in the cryogenic trap.

4.3.2.4 Open the helium line to the desorption chambers.

4.3.2.5 Adjust the helium flow under each chamber to approximately 15 mL/min.

4.3.2.6 Place the cartridges in the desorption chambers with the bottom end down (see Figure 1).

4.3.2.7 After all the cartridges are in place recheck the flows from each chamber.

CAUTION

To avoid contamination of the Tenax, ensure that helium is flowing through every cartridge.

4.3.2.8 Desorb the Tenax cartridges for five hours.

4.3.2.9 Refill the cryogenic trap with liquid nitrogen every hour, or when the level of liquid nitrogen is less than one-third full.

CAUTION

If liquid nitrogen in the trap is depleted all the impurities trapped in the line will be transported to the Tenax.

4.3.2.10 Record all pertinent information on the Tenax Cleanup Worksheet (see Figure 2) for the specific Tenax batch.

4.3.2.11 Recheck the helium flow every two hours and before removing the cartridges.

4.3.2.12 Remove each cartridge with a pair of tweezers and immediately place the hot cartridge in a Kimax culture tube.

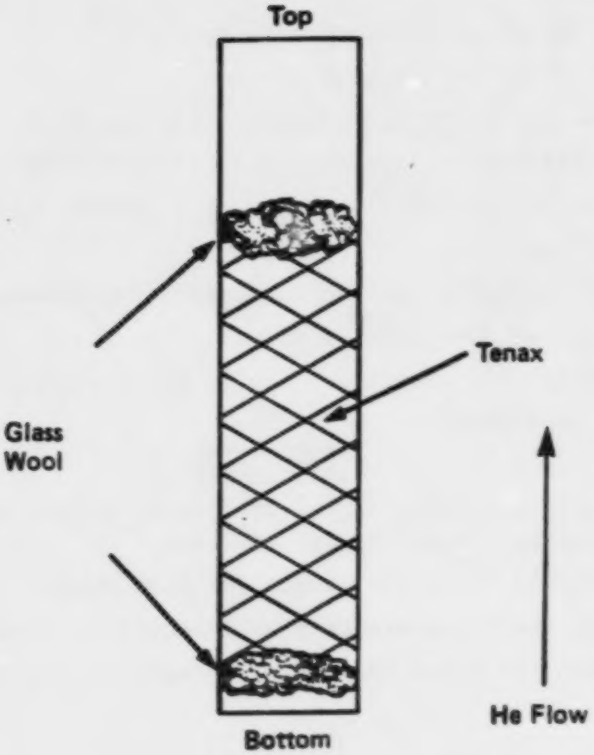
Top
Tenax
Glass
Wool
He Flow
Bottom

Figure 1. Tenax cartridge.

TENAX CLEANUP WORKSHEET

Tenax Batch No. ____________________ Projected Use__________________

Virgin/Recycled. Recycled Source ________________ No. of Cartridges _____________

Extraction

Number of Soxhlet Units (circle one): 1 2 3 4 5

Methanol Extraction: Date (Hours)

Siphon Rate

Pentane Extraction: Date (Hours)

Siphon Rate

Drying

Nitrogen Chamber: Date (Hours)

Approx. Flowrate

Vacuum Oven: Date (Hours)

Pump Trap

Cooldown (Hours)

N_2 Vent Thru Act. C

Sieving/Packing

Sieve (40/60) Date:

Packing Date(s):

Cleanup

Teflon Septum; Date:

Teflon Liner; Date:

Figure 2. Tenax cleanup worksheet - Form A

Page 2 of 2

TENAX CLEANUP WORKSHEET

Desorption

Lot: A B C D E F G H

Date:

Hours:

Temp.:

Liq. N_2:

He Flow:

GC Check

Date:

GC Identifier No.

No. of Cart.

Peaks >30% fs:

Peaks <30% fs:

OK/NG:

Redesorption

Lot:

Date:

Reason:

GC Check:

Figure 2. Tenax cleanup worksheet - Form B.

4.3.2.13 Seal the tube.

4.3.2.14 Label the screw cap with the Tenax batch number and the culture tube with the desorption date.

4.3.2.15 Store the culture tubes in the "clean" room in one gallon paint cans.

4.4 Background Check

Ten percent of the cartridges from each batch are analyzed for background contamination using GC/FID. The ten percent selected must include cartridges from each desorption unit and from every desorption period of that batch.

4.4.1 Analyze the Tenax cartridges using a Varian 3700 GC equipped with a thermal desorption injection unit and a flame ionization detector, and interfaced to a Varian CDS III integrator.

4.4.2 Select one cartridge from each desorption period and from every unit.

4.4.3 Analyze each cartridge at least 24 hours after desorption.

NOTE: Studies have shown evidence for the migration of contaminants from the interior of Tenax beads as a function of time.

4.4.4 Analyze the cartridges using the following instrumental conditions.

4.4.4.1 Colum: fused silica capillary column (30 m X 0.25 μm) Liq. phase DB1; or equivalent.

4.4.4.2 Detector temperature: 300°C

4.4.4.3 Injector temperature: 280°C

4.4.4.4 Desorption unit temperature: 260°C

4.4.4.5 Temperature program: 30°C (5 min), 4°C/min to 220° (10 min)

4.4.4.6 Attenuation: 128×10^{-12} (or as required for adequate recorder response)

4.4.4.7 Desorption time: 8 min

4.4.4.8 Helium carrier flow: 1.2 mL/min

4.4.4.9 H_2 flow to FID ~35 mL/min

4.4.4.10 Air flow to FID: ~300 mL/min

4.4.4.11 Make-up gas: ~30 mL/min

4.4.4.12 Recorder chart speed: 1 cm/min

4.4.5 Examine the chromatographic retention window for each target. The window is defined as the mean retention time (5 replicates) ± 1σ.

NOTE: The chromatographic retention window for each target compound is determined prior to the cartridge evaluation from the analysis of cartridges loaded with known amounts of the compounds of interest (see SOP-630-001 and SOP-630-002).

4.4.6 In your notebook, record the total area counts for each window.

4.4.7 Compare the area counts to the historical area count data corresponding to the limits of detection (LOD) for each compound as determined by GC/MS analysis (see Table 1).

NOTE: The historical area count data is obtained prior to the cartridge evaluation by loading each compound at the GC/MS limit of detection, and determining its respective area by GC/FID analysis.

4.4.8 If no peaks or only one peak falls within a window of study, the set of cartridges is accepted provided that the area of the peak does not exceed three times the LOD.

4.4.9 If more than one peak falls within the windows of study, but the areas are lower than those corresponding to the limits of detection, then the set of cartridges is acceptable.

4.4.10 If the areas of more than one peak exceed the area corresponding to the LOD for that window, the set of cartridges is rejected and must be redesorbed.

4.4.11 Cartridges may be deemed acceptable if analysis by GC/MS indicates clean windows, regardless of GC/FID results. Task Leader approval is required for acceptance of such cartridges (i.e., those deemed unacceptable by GC/FID, but acceptable by GC/MS).

Table 1. LIMITS OF DETECTION FOR TEAM TARGET COMPOUNDS

Compound	LOD (ng/cartridge)
Bromodichloromethane	10
Chlorodibromomethane	10
Chloroform	6
1,1,1-Trichloroethane	7
Benzene	3
Carbon tetrachloride	10
Tetrachloroethylene	10
Styrene	4
m- or p-Dichlorobenzene	4
Ethylbenzene	3
Xylene, o, m, or p	3
Trichloroethylene	14
Chlorobenzene	4
Tetrachloroethane (isom)	5
1,2-Dichloroethane	7
n-Decane	5
n-Undecane	5
n-Dodecane	5

Source: Historical LOD data from Finnigan 3300.

4.4.12 After a sample has been analyzed, label the desorption unit lot from which it was drawn according to the following criteria.

4.4.12.1 If the set of cartridges is acceptable label it "Ready to be packed".

4.4.12.2 If the cartridges are rejected, label the can "To be redesorbed" "Date".

4.4.13 Quality Assurance for the Background Check.

4.4.13.1 Every three days, run a control cartridge loaded with the GC/MS LOD amounts.

4.4.13.2 Determine the chromatographic retention windows for each target compound and the corresponding area counts.

4.4.13.3 Compare the retention window and the corresponding area with the historical data.

4.4.13.4 If an historical data base does not exist, create one after loading three cartridges with the appropriate amounts of target compounds.

4.4.13.5 If any major alterations are made to the GC (i.e., replacement of the thermal desorption/injection unit, cleaning of the detector; replacement of the column), create a new data base.

4.4.13.6 If the analysis of the control cartridge indicates that two or less compounds are out-of-control, begin the background check.

NOTE: A compound is considered out-of-control when its retention window deviates from the mean value by more than two standard deviations, and its mean area counts by more than three standard deviations.

4.4.13.7 If more than two compounds are out-of-control, load a second cartridge and analyze it.

4.4.13.8 If the second cartridge is in control, proceed with the background check.

4.4.13.9 If it is out-of-control, troubleshoot the instrument before proceeding with the background check.

RTI/ACS-SOP-322-001
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Revision 0

Standard Operating Procedure for the Cleanup of Water Collection Bottles

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Standard Operating Procedure for the Cleanup
# of Water Collection Bottles
RTI/ACS-SOP-322-001

1.0 PRINCIPLE

The amber glass bottles, caps, and liners used for the collection of water samples must be thoroughly cleaned prior to use. Preparation involves washing, baking at high temperatures, and capping the bottles with Teflon®-lined caps.

2.0 MATERIALS

2.1 Two ounce narrow mouth, amber glass bottles
2.2 Foil-lined phenolic resin caps
2.3 Teflon® cap liners
2.4 Methanol, pesticide grade
2.5 Forceps

3.0 PROCEDURE

3.1 Caps and Liners

3.1.1 Sonicate Teflon® cap liners in methanol for approximately fifteen minutes.
3.1.2 Pour off methanol and rinse twice with deionized water.
3.1.3 Dry cap liners in a clean oven for 2 to 4 hours at a temperature not exceeding 150°C.
3.1.4 Remove cap liners from the oven and store in a clean glass jar sealed with a foil-lined cap.
3.1.5 Using forceps, prepare caps by inserting the Teflon® liner into the foil-lined caps.

NOTE: This operation should be performed just prior to capping the water bottles in order to minimize contamination of the cap liners. Use only forceps to handle the cap liners.

3.2 Water Bottles

3.2.1 Wash the water bottles thoroughly, then rinse with deionized water.

3.2.2 Air dry all bottles in an inverted position.

3.2.3 Bake out bottles in a high temperature oven, approximately 900°F for two hours.

3.2.4 Allow the bottles to cool to room temperature.

3.2.5 Inspect all bottles for damage and discard broken, chipped, or poorly-formed bottles.

CAUTION

Small chips or cracks in the bottles prevent the caps forming a proper seal which may result in the loss of volatile compounds from the sample during storage.

3.2.6 Store the capped bottles in a clean area until needed.

RTI/ACS-SOP-331-001
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Revision 1

Standard Operating Procedure for the Collection of Personal Air Samples

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Standard Operating Procedure for the Collection of Personal Air Samples

RTI/ACS-SOP-331-001

1.0 PRINCIPLE

Personal air samples shall be collected during the entire period that the participant is involved in the study. The collections provide a representative sample of the air which a participant has been exposed to during the sampling period. A sampler shall be worn by the participant for the entire sampling period as they go about their normal daily activities.

2.0 MATERIALS

2.1 DuPont P-125A Constant Flow Samplers
2.2 Bubble flow meter, 25 mL
2.3 Stopwatch/Calculator
2.4 Tenax sampling trains
2.5 Glass fiber filters, 25 mm (Gelman Sciences)
2.6 Forceps
2.7 Kimwipes®
2.8 Sampling vests
2.9 Mercury thermometer

3.0 PROCEDURE

Two personal air samples of approximately equal volume shall be collected during a 24-hour sampling period. The participant shall wear on their person a personal sampler containing one Tenax cartridge. At night or during unusual circumstances, the sampler may be removed and placed in close proximity to the participant.

3.1 Personal Air Sampling Schedule

3.1.1 Refer to the Sample Collection Schedule (Figure 1) and determine what samples are to be collected from each participant and the Tenax batch numbers for those samples.

3.1.2 Prepare the labels and Chain-of-Custody (COC)/Field Sampling Protocol (SP) sheets for all of the samples to be collected for the participant (see Figures 2 and 3).

3.1.3 Remove and label the appropriate number of Tenax cartridges required.

3.1.4 Store all of the labelled Tenax cartridges in the Tenax storage area until needed.

NOTE: If more than one Tenax batch number has been assigned per matrix, use Tenax from the same batch for all the field and duplicate samples of a given participant.

3.2 Preparation of Sampling Equipment

3.2.1 At least 30 minutes prior to leaving for the first scheduled appointment, calibrate the DuPont P-125A samplers. See RTI/ACS-SOP-361-001, Calibration of DuPont P-125A Constant Flow Samplers, for details.

NOTE: The ideal air sample volume is 20 ± 15% liters (17-23 L). A pumping rate should be used which will give a sample volume in this range over the collection period.

3.2.1.1 If the anticipated collection time is between approximately 11 and 13 hours, any flow rate in the range of 25-30 mL/min will be adequate.

3.2.1.2 If the collection period will be less than 11 hours, use a pump with a correspondingly higher flow rate (30-60 mL/min). Do not use a flow rate less than 25 mL/min.

3.2.2 Assemble the sampling train, less the glass fiber filter and Tenax cartridge (see Figure 4).

3.2.3 Collect Kimwipes®, forceps, glass fiber filters, thermometer, and sampling vests.

City ____________ (Season) - PERSONAL AIR (AV) ____________

Batch # ____________

____

		Samples Collected		
Participant Number	Field	Duplicates	QA	Expose QC Set
21476-7	2			
21477-5	2	2		1
21478-3	2			
21479-1	2			
21480-9	2			
21481-7	2			
21482-5	2	2		
21483-3	2			
21484-1	2		2	
21485-8	2			
21486-6	2			1
21487-4	2	2		
21488-2	2			
21489-0	2			
21490-8	2			
21491-6	2			
21492-4	2	2		
21493-2	2			
21494-0	2		2	1
21495-7	2			
21496-5	2			
21497-3	2	2		
21498-1	2			
21499-9	2			
21500-4	2			

Figure 1. Sample collection schedule (example).

FIELD SAMPLING PROTOCOL SHEET - TEAM STUDY

DATE: ______________________

SAMPLE CODE: INAIR 111-5 VO-F1

AIR SAMPLING

Pump Model: P125A Serial Number: 02

Time (Initial): 16:00 Flow Rates (Initial): 25.4

(Final): 9:00 (Final): 25.6

Total Time: 1020 min Avg. Flow Rate: 25.5

Total Volume Sampled: 26.0 L

Temperature (°F): 77/79 = 78°F

BREATH SAMPLING

Meter Readings Final: ________ ft^3 Time of Day: ______________

Initial: ________ ft^3 Temperature: ______________ °F

Total: ________ ft^3 Duration of Exhalation __min__sec

Correction Factor: X ________ Pump No.: ______________

Volume/Cartridge: ______________ L (1 ft^3 = 28.3 L)

DRINKING WATER, BLOOD, URINE, MILK SAMPLING

Time of Day: ______________ Source (water only) __________
(*e.g.* ground, city, etc.)

Amount Collected: __________

FOOD SAMPLING

Time Period During Which Food was Consumed: ______________

Time of Collection: __________

Container	Container Type	Description of Food Type
(1)		
(2)		
(3)		

GENERAL REMARKS

Figure 2. Field Sampling Protocol Sheet (Front)

CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute, Analytical Sciences Division
Chemistry and Life Sciences Group, Research Triangle Park, NC 27709

SAMPLE CODE INAIR 111-5 VO-F1	**TENAX BATCH:** 61	**VOLUME COLLECTED:** 26.0
SAMPLE TYPE: Tenax	**TEMPERATURE (°F)** 78	**VOLUME ANALYZED:**

RELINQUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	JS	1600	7/11/83	Tenax Exposed
JS	JS	900	7/12/83	Tenax Collected

Figure 3. Chain-of-Custody Sheet (Back)

3.2.4 Remove the Tenax cartridges from the Tenax storage area and place into a field collection can.

NOTE: Remove only those cartridges which will be exposed during the immediate appointment. The additional cartridges should remain in the Tenax storage area until needed.

3.3 Collection of the Initial Personal Air Sample

*During the first appointment with the participant, a fully charged DuPont sampler shall be used for the collection of the initial field sample. The following operations are performed at the participant's residence, with the exception that the pumps may be calibrated in the house, van or workroom. Similarly, pump number and flow rate may be recorded in the workroom, the van, or the participant's house.

3.3.1 Transport the necessary sampling equipment to the participant's residence (see Section 3.2.2 to 3.2.4).

3.3.2 Attach the sampling train to the inlet (top) barb of the DuPont sampler.

3.3.3 Install one glass fiber filter in the top filter holder of the sampling train.

NOTE: Filters are replaced at the beginning of each new 24-hour sampling period or more frequently if the filters appear damaged or soiled.

3.3.4 Record the sampler number, flow rate, and time on the sample SP sheet.

3.3.5 Remove the Tenax cartridge from the field collection can and reseal the can.

3.3.6 Using forceps, remove the top pad of glass wool from the culture tube and place it on a clean Kimwipe®.

3.3.7 Using a clean Kimwipe®, remove the Tenax cartridge from the culture tube.

3.3.8 Install the Tenax cartridge in the sampling train (see Figure 4).

CAUTION

Do not allow the Tenax cartridge to touch the hands or other material. Contamination may result. Install the cartridge in the proper orientation with the void end nearest the DuPont sampler.

3.3.9 Using forceps replace the glass wool pad.

3.3.10 Return the empty culture tube to the field collection can and reseal the can.

3.2.4 Remove the Tenax cartridges from the Tenax storage area and place into a field collection can.

NOTE: Remove only those cartridges which will be exposed during the immediate appointment. The additional cartridges should remain in the Tenax storage area until needed.

3.3 Collection of the Initial Personal Air Sample

*During the first appointment with the participant, a fully charged DuPont sampler shall be used for the collection of the initial field sample. The following operations are performed at the participant's residence, with the exception that the pumps may be calibrated in the house, van or workroom. Similarly, pump number and flow rate may be recorded in the workroom, the van, or the participant's house.

3.3.1 Transport the necessary sampling equipment to the participant's residence (see Section 3.2.2 to 3.2.4).

3.3.2 Attach the sampling train to the inlet (top) barb of the DuPont sampler.

3.3.3 Install one glass fiber filter in the top filter holder of the sampling train.

NOTE: Filters are replaced at the beginning of each new 24-hour sampling period or more frequently if the filters appear damaged or soiled.

3.3.4 Record the sampler number, flow rate, and time on the sample SP sheet.

3.3.5 Remove the Tenax cartridge from the field collection can and reseal the can.

3.3.6 Using forceps, remove the top pad of glass wool from the culture tube and place it on a clean Kimwipe®.

3.3.7 Using a clean Kimwipe®, remove the Tenax cartridge from the culture tube.

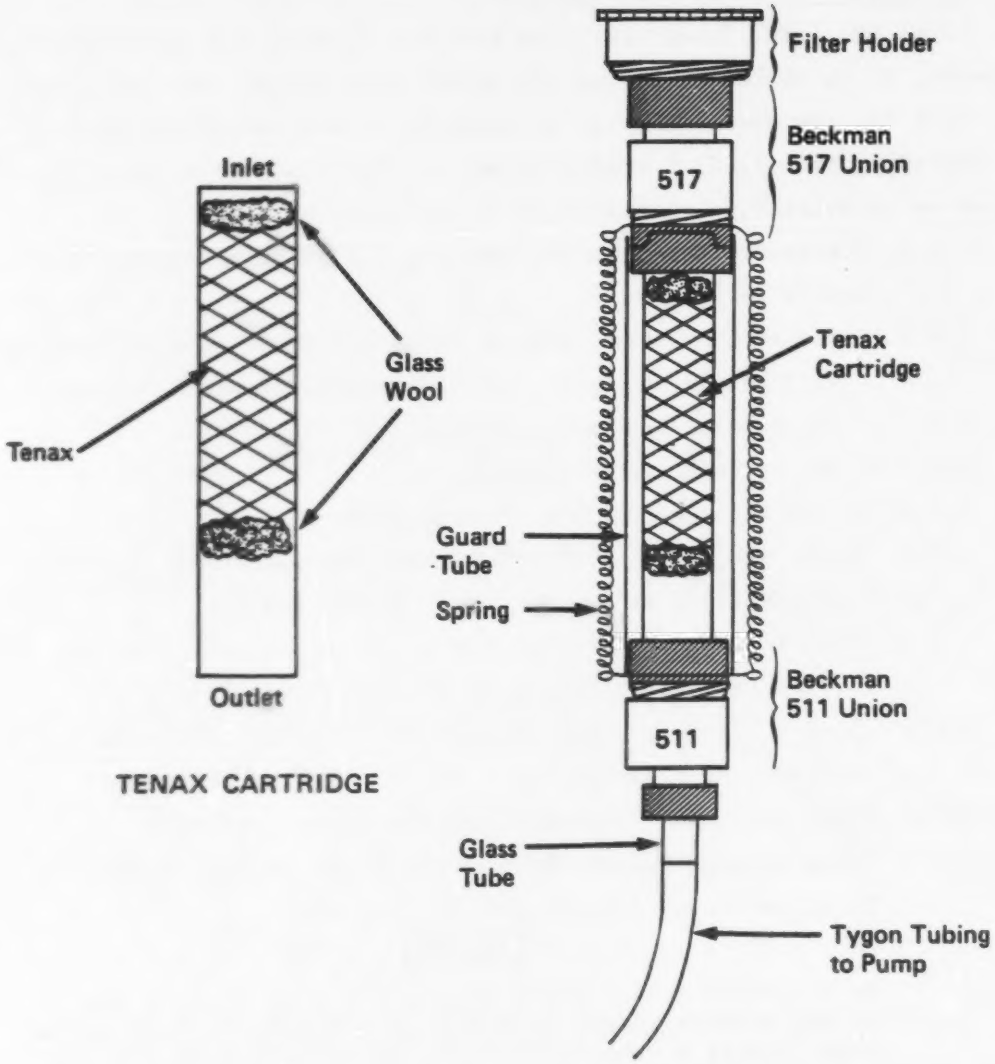
3.3.8 Install the Tenax cartridge in the sampling train (see Figure 4).

CAUTION

Do not allow the Tenax cartridge to touch the hands or other material. Contamination may result. Install the cartridge in the proper orientation with the void end nearest the DuPont sampler.

3.3.9 Using forceps replace the glass wool pad.

3.3.10 Return the empty culture tube to the field collection can and reseal the can.


Inlet
Glass Wool
Tenax
Outlet
Filter Holder
Beckman 517 Union
517
Tenax Cartridge
Guard Tube
Spring
Beckman 511 Union
511
Glass Tube
Tygon Tubing to Pump

TENAX CARTRIDGE

SAMPLING TRAIN

Figure 4. Tenax Cartridge and Sampling Train

3.3.11 Instruct the participant to wear the sampler at all times when possible.

NOTE: If the vest is to be used, it should be worn as depicted in Figure 5. The sampler may be attached to the belt or waistband if the sampling train is clipped near the breathing zone.

3.3.12 Return the field collection can to the Tenax storage area.

3.4 Collection of Subsequent Samples

After the first Tenax cartridge has been exposed for approximately 12 hours, it is collected during the second appointment, and the final flow rate for the DuPont sampler is measured. Once the flow rate corresponding to a 17-23 L sample volume for the next collection period is calculated, a second Tenax cartridge is exposed.

3.4.1 Transport the necessary sampling equipment to the participant's residence.

3.4.1.1 The empty culture tube in which the first sample cartridge will be placed and the next unexposed sample cartridge.

3.4.1.2 The bubble flow meter and stopwatch/calculator.

3.4.1.3 The forceps and Kimwipes®.

3.4.2 Obtain the sampler from the participant.

3.4.3 Remove the empty culture tube and the next sample cartridge from the field collection can. Reseal the can.

3.4.4 Using forceps, remove the top pad of glass wool from the empty culture tube and place it on a clean Kimwipe®.

3.4.5 Using a clean Kimwipe®, remove the Tenax cartridge from the sampling train and note the time on the SP sheet.

3.4.6 Place the Tenax cartridge into the culture tube.

3.4.7 Using forceps, place the glass wool pad on top of the Tenax cartridge and cap the culture tube.

CAUTION

Do not allow excess glass wool to lie over the top edge of the culture tube. This will prevent the cap and septum from forming a proper seal with the culture tube possibly resulting in the contamination of the sample during storage.

3.4.8 Place the collected sample into the field collection can and reseal the can.

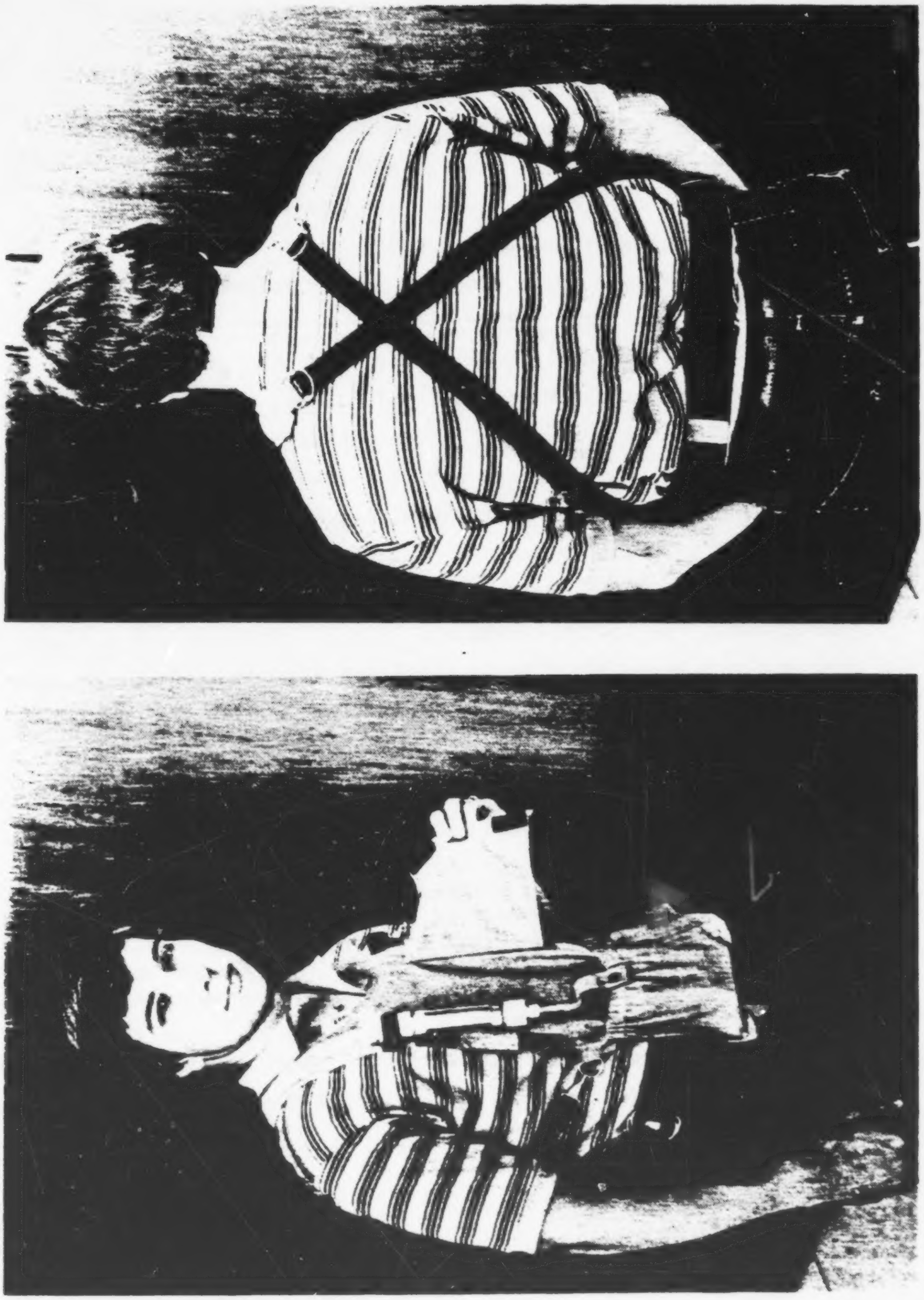


Figure 5. Sampler and Vest

3.4.9 Attach the bubble flow meter to the exhaust, side, barb of the DuPont sampler.

3.4.10 Measure the flow rate three times and calculate the mean flow rate.

3.4.11 Record the time at which the sample was removed from the train, and the mean flow rate on the SP sheet for the sample.

3.4.11.1 If the mean flow rate will give an air volume of 17-23 L during the next collection period, use the same sampler for the collection of the next sample.

3.4.11.2 If the mean flow rate will not give an air volume of 17-23 L during the next collection period, reset the flow rate and recalibrate the sampler using the procedures described in RTI/ACS-SOP-361-001, Calibration of DuPont P-125A Constant Flow Samplers or use a different sampler which has been set at the proper flow rate.

3.4.12 Using the unexposed Tenax cartridge, repeat steps 3.3.2-3.3.11 for the collection of the next sample.

3.4.13 Enter the pump number, date, time and new flow rate on the SP sheet for the next sample.

3.4.14 Return the collected sample to the Tenax storage area.

3.4.15 Store the collected sample in a can designated for personal air samples only.

3.4.16 Store the collected sample in chronological order until the can contains no more than 17 cartridges.

NOTE: In order to facilitate the final QA check and packing of the cartridges prior to shipment, the collected cartridges should be stored in numbered cans as the are collected. As a result, the cartridges are grouped in order of their collection dates as well as by sets. Once a can is full the COC sheets may be easily matched with their corresponding samples and grouped together by can number.

3.4.17 Complete the COC sheet for the collected sample including a quality assurance check by another field staff member.

NOTE: A quality assurance check involves a complete check of all information listed on the COC sheet including all calculations, initials, dates, labels and times.

3.4.18 Complete any information needed on the COC sheet for the newly exposed sample.

3.5 Collection of the Final Sample

The collection of the final sample is similar to the collection of any previous samples except the DuPont sampler is returned to the workroom and recharged.

3.5.1 Transport the necessary sampling equipment to the sample site.

3.5.1.1 The empty culture tube in which the exposed Tenax cartridge will be placed.

3.5.1.2 The bubble flow meter and stopwatch.

3.5.1.3 The forceps and Kimwipes®.

3.5.2 Collect the sampler from the participant.

*3.5.3 Repeat steps 3.4.3-3.4.11 for the collection of the final sample.

NOTE: Pump calibration must be performed within one hour after the sample collection period.

3.5.4 Place the running DuPont sampler in the workroom allowing it to discharge.

*3.5.5 Turn off the samplers and place them on the battery charger at the full charge rate. After 16 hours on full charge, the samplers may be removed and stored for several days or switched to trickle charge and left on the chargers indefinitely.

NOTE: A charing time of 16 hours at the full charge rate is recommended, but not mandatory, in order to insure a fully chargled sampler for the next day. Pumps used overnight, replaced in the morning, and needed that afternoone may be charged during the time available.

3.5.6 Return the collected sample to the Tenax storage area and store as described in Section 3.4.16.

3.5.7 Complete the COC sheet for the collected sampling including a quality assurance check by another field staff member.

RTI/ACS-SOP-331-002
April 1984
Revision 1

Standard Operating Procedure for the Collection of Fixed Site Air Samples

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Vice-President, RTI/ACS Date

Standard Operating Procedure for the Collection of Fixed Site Air Samples

RTI/ACS-SOP-331-002

1.0 PRINCIPLE

The collection of a fixed site air sample will provide a sample of the ambient air to which the participant is exposed.

2.0 MATERIALS

2.1 DuPont P-125A Constant Flow Sampler
2.2 Bubble flow meter, 25 mL
2.3 Stopwatch/calculator
2.4 Tenax sampling trains
2.5 Glass fiber filters, 25 mm (Gelman Sciences)
2.6 Metal fixed site boxes
2.7 Forceps
2.8 Kimwipes®

3.0 PROCEDURE

Two fixed site air samples of approximately equal volume will be collected during the 24-hour sampling period. If possible, a fixed site sample shall be collected at the residence of the first participant from a sample "segment" scheduled for sample collection. The procedures for the collection of field, duplicate, and Quality Assurance samples are identical.

3.1 Assignment of Fixed Site Samples

Fixed site air samples are not collected for every participant. Only one sample set is scheduled for collection in every sample segment containing a participant. In order to insure the collection of the required fixed site samples, the first partiicpant scheduled for sampling in a given segment is usually assigned a fixed site sample. Subsequent participants in a previously unsampled segment will be scheduled for fixed site air collections.

3.1.1 Refer to the Appointment Control Sheet (Figure 1) and note the sample segment number assigned to the participant.

3.1.2 Refer to the Sample Collection Schedule for fixed site air samples (Figure 2) and determine if the sample segment number for the participant has been previously sampled.

NOTE: Once a fixed site sample has been collected in a sample segment, that segment number is then listed on the Sample Collection Schedule under the appropriate column.

3.1.2.1 If the participant's segment number is listed on the Sample Collection Schedule (column 2), no fixed site sample shall be assigned to that participant.

3.1.2.2 If the participant's segment number is not listed on the Sample Collection Schedule (column 2), the participant shall be assigned the next set of fixed site samples.

3.1.3 From the Sample Collection Schedule, determine if any duplicate of Quality Assurance samples are to be collected with the assigned fixed site field samples.

3.1.4 From the Sample Collection Schedule, determine what Tenax batch number is to be used for the fixed site samples.

NOTE: Tenax batch numbers are predetermined for each matrix and are specific for that matrix.

3.1.5 Prepare the labels and Chain-of-Custody (COC)/Field Sampling Protocol (SP) sheets for all the samples which will be collected for the participant (see Figures 3 and 4).

3.1.5.1 Enter the Tenax batch number for each respective sample on the COC sheet.

3.1.5.2 Enter the sample segment number and participant number on the Sample Collection Schedule in columns 2 and 3, respectively.

APPOINTMENT CONTROL SHEET

F.I. Name ______________________________
Date ___/___/___ Day of Week ______________________
City ______________________

1) Site # ___ Segment # ___ ___
HU # ___ ___ ___ Participant # ___ ___
Name ______________________________
Address ______________________________
Telephone # ______________

Evening Appointment * ______________________
Morning Appointment ** ______________________
Afternoon Appointment *** ______________________

2) Site # ___ Segment # ___ ___
HU # ___ ___ ___ Participant # ___ ___
Name ______________________________
Address ______________________________
Telephone # ______________

Evening Appointment * ______________________
Morning Appointment ** ______________________
Afternoon Appointment *** ______________________

3) Site # ___ Segment # ___ ___
HU # ___ ___ ___ Participant # ___ ___
Name ______________________________
Address ______________________________
Telephone # ______________

Evening Appointment * ______________________
Morning Appointment ** ______________________
Afternoon Appointment *** ______________________

Information given to Chemists at ________ p.m.[†] on ________,
___/___/81. Person spoken to ______________________.

* Schedule at half hour intervals between 7 and 9 p.m.
** Schedule at half hour intervals between 6 and 9 a.m.
*** Schedule at one hour intervals between 4 and 6 p.m.

[†] Call between 12:00 noon and 2:00 p.m. at least 24 hours before the first appointment.

Figure 1. Appointment Control Sheet

Batch: ____________________ Site (Season) — Fixed Air

Fixed Air No.	Seg. No.	Part No.	Field	Duplicate	QA	Expose QC Set
01			2			1
02			2	2		
03			2			
04			2		2	
05			2			

Figure 2. Sample Collection Schedule (Example)

FIELD SAMPLING PROTOCOL SHEET - TEAM STUDY

DATE: ______________________

SAMPLE CODE: INAIR 111-5 XV-F1

AIR SAMPLING

Pump Model: P125A Serial Number: 01

Time (Initial): 15:00 Flow Rates (Initial): 25.4

(Final): 8:00 (Final): 25.6

Total Time: 1020 min. Avg. Flow Rate: 25.5

Total Volume Sampled: 26.0 L

Temperature (°F): ______________

BREATH SAMPLING

Meter Readings Final: ________ ft^3 Time of Day: ______________

Initial: ________ ft^3 Temperature: ______________ °F

Total: ________ ft^3 Duration of Exhalation __min__sec

Correction Factor: X ________ Pump No.: ______________

Volume/Cartridge: ______________ L (1 ft^3 = 28.3 L)

DRINKING WATER, BLOOD, URINE, MILK SAMPLING

Time of Day: ______________ Source (water only) ______________
(*e.g.* ground, city, etc.)

Amount Collected: ______________

FOOD SAMPLING

Time Period During Which Food was Consumed: ______________

Time of Collection: ______________

Container	Container Type	Description of Food Type
(1)		
(2)		
(3)		

GENERAL REMARKS

Figure 3. Chain-of-Custody Sheet (Front)

CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute, Analytical Sciences Division
Chemistry and Life Sciences Group, Research Triangle Park, NC 27709

SAMPLE CODE INAIR 111-5 XV-F1	**TENAX BATCH:** 61	**VOLUME COLLECTED:** 26.0
SAMPLE TYPE: Tenax	**TEMPERATURE (°F)**	**VOLUME ANALYZED:**

RELIN-QUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	JS	1500	5/12/83	Tenax Exposed
JS	JS	800	5/13/83	Tenax Collected
JS				

Figure 4. Chain-of-Custody Sheet (Back)

3.1.6 From the batch of Tenax designated for fixed site air, remove and label the appropriate number of cartridges.

NOTE: If more than one Tenax batch number has been assigned per matrix, try to use the same batch numbers to all of the field and duplicate samples of a given participant.

3.1.7 Store all the Tenax cartridges in the Tenax storage area until needed.

3.2 Preparation of Sampling Equipment

3.2.1 At least 30 minutes prior to leaving for the first scheduled appointment, calibrate the DuPont P-125A Constant flow samplers. (See RTI/ACS-SOP-361-001, Calibration of DuPont P-125A Constant Flow Samplers for details.)

NOTE: The ideal air sample volume is 20 ± 15% liters (17-23 L). A pumping rate should be used which will give a sample volume in this range over the collection period.

3.2.1.1 If the anticipated collection time is between approximately 11 and 13 hours, any flow rate in the range of 25 to 30 mL/min will be adequate.

3.2.1.2 If the collection period will be less than 11 hours, use a pump with a correspondingly higher flow rate (30-60 mL/min). Do not use flow rate less than 25 mL/min.

3.2.2 Take one metal fixed site box for each participant scheduled for the collection of fixed site samples. Each box holds two complete sampling units (see Figure 5) and protects them from inclimate weather.

3.2.3 Assemble the sampling train without the glass fiber filter and Tenax cartridge (see Figure 6).

3.2.4 Place Kimwipes®, forceps, and glass fiber filters in the sampling equipment box carried to the participant's residence.

3.2.5 Remove the Tenax cartridges from the Tenax store area and place into a field collection can.

NOTE: Remove only those cartridges which will be exposed during the immediate appointment. The additional cartridges should remain in the storage area until needed.

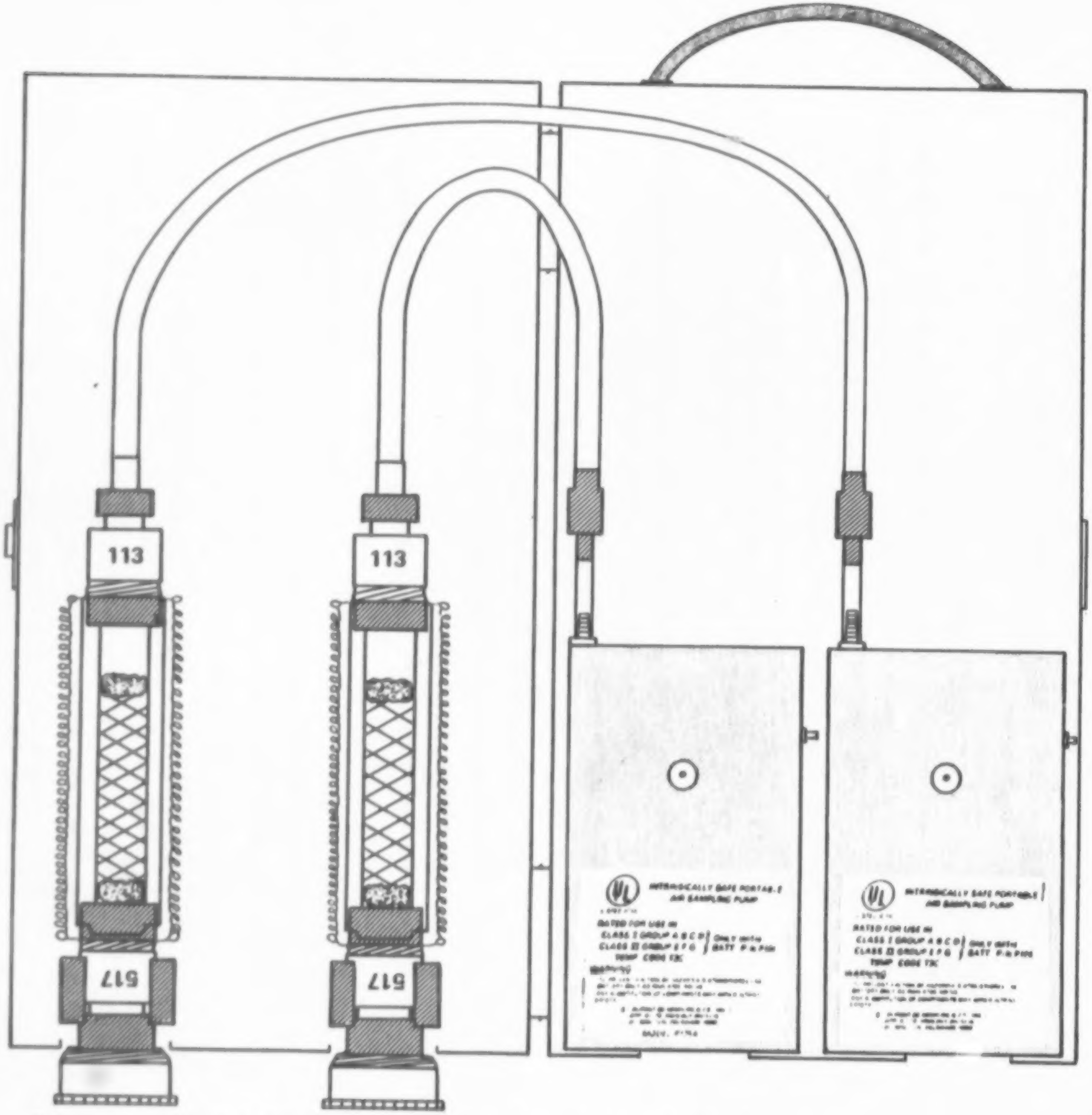
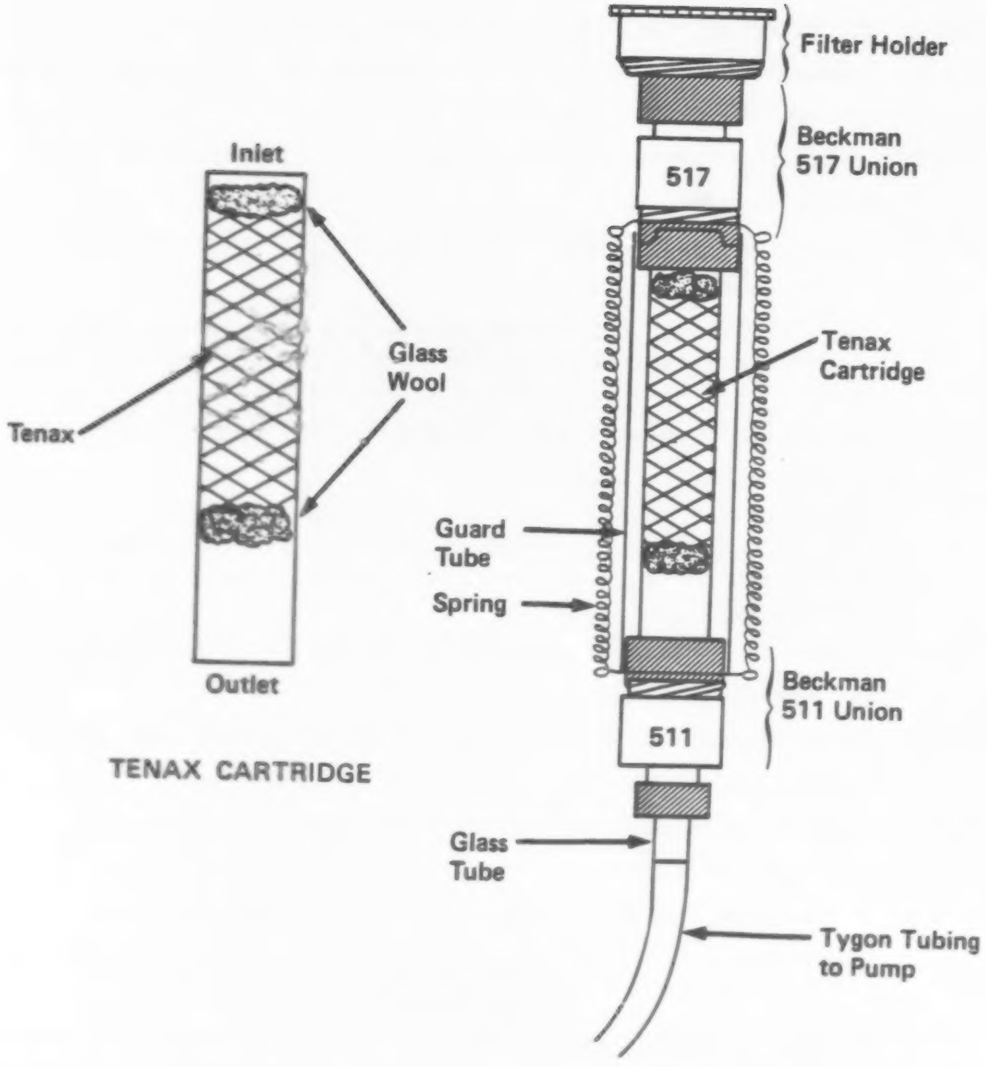

113
113
517
517

Figure 5. Fixed Site Samplers In Box (Open-Internal View)


Inlet
Glass Wool
Tenax
Outlet
Filter Holder
Beckman 517 Union
517
Tenax Cartridge
Guard Tube
Spring
Beckman 511 Union
511
Glass Tube
Tygon Tubing to Pump

TENAX CARTRIDGE

SAMPLING TRAIN

Figure 6. Tenax Cartridge and Sampling Train

3.3 Exposure of the Initial Fixed Site Sample

* During the first appointment with a participant, a fully charged DuPont sampler shall be used for the collection of the initial field sample. The following operations shall be performed at the participant's residence, with the exception that the pumps may be calibrated in the house, van or workroom. Similarly, pump number and flow rate may be recorded in the workroom, the van, or the participant's house.

3.3.1 Transport the necessary sampling equipment to the participant's residence (see Sections 3.2.2 to 3.2.5).

3.3.2 Attach the sampling train to the inlet (top) barb of the DuPont sampler.

3.3.3 Install one glass fiber filter in the top filter holder of the sampling train.

NOTE: Filters are replaced at the beginning of each new 24-hour sampling period or during the appointment when Tenax cartridges are changed if the filter is damaged or soiled.

*3.3.4 Record the sampler number, flow rate, and time on the SP sheet for the sample.

NOTE: Do not record the temperature for fixed-site air samples. Ambient air temperature, humidity, wind speed and direction records should be obtained from a local weather service.

3.3.5 Remove the Tenax cartridge from the field collection can and reseal the can.

3.3.6 Using forceps, remove the top pad of glass wool from the culture tube and place it on a clean Kimwipe®.

3.3.7 Using a clean Kimwipe®, remove the Tenax cartridge from the culture tube.

3.3.8 Install the Tenax cartridge in the sampling train (see Figure 6).

CAUTION

Do not allow the Tenax cartridge to touch the hands or other material. Contamination may result. Install the cartridge in the proper orientation with the void end nearest the DuPont sampler (see Figure 6).

3.3.9 Using forceps replace the glass wool pad.

3.3.10 Return the empty culture tube to the can and reseal the can.

3.3.11 Place the sampler into the metal fixed site box as depicted in Figure 5.

3.3.12 Place the fixed site box outside of the participant's residence. The location should meet the following criteria:

- 3.3.12.1 Exterior location.
- 3.3.12.2 Secure location.
- 3.3.12.3 Away from exterior vents, air conditioners, fans, or areas which may compromise the integrity of the sample.
- 3.3.12.4 Positioned at approximately eye level.

3.3.13 Return the field collection can to the Tenax storage area.

3.3.14 Complete the sample SP sheet including a diagram showing the position of the fixed site box relative to the participant's residence.

3.4 Collection of Subsequent Samples

After the exposure of the first Tenax cartridge for approximately 12 hours, the cartridge is collected and the final flow rate for the DuPont sampler is determined. Once the flow rate corresponding to a 17-23 L sample volume for the next collection period is calculated, a second Tenax cartridge is exposed.

3.4.1 Transport the necessary sampling equipment to the sample site.

- 3.4.1.1 The empty culture tube in which the first sample cartridge will be placed and the next unexposed sample cartridge.
- 3.4.1.2 The bubble flow meter and stopwatch/calculator.
- 3.4.1.3. The forceps and Kimwipes®.

3.4.2 Remove the sampler from the fixed site box.

NOTE: In order to eliminate any possible confusion of sampling trains and DuPont samplers, particularly in the event duplicate samplers are present, do not remove the sampling trains from the samplers at this point.

3.4.3 Remove the empty culture tube and the next sample cartridge from the field collection can. Reseal the can.

3.4.4 Using forceps, remove the top pad of glass wool from the empty culture tube and place it on a clean Kimwipe®.

3.4.5 Using a clean Kimwipe® remove the Tenax cartridge from the sampling train.

3.4.6 Place the exposed cartridge into the empty culture tube.

3.4.7 Using forceps, place the glass wool pad on top of the Tenax cartridge.

CAUTION

Do not allow excess glass wool to lie over the top edge of the culture tube. This will prevent the cap and septum from forming a proper seal with the culture tube possibly resulting in contamination of the sample during storage.

3.4.8 Place the collected sample in the field collection can, reseal the can and record on the SP sheet the time the cartridge was removed from the sampler.

3.4.9 Attach the bubble flow meter to the exhaust (side) barb of the DuPont sampler.

3.4.10 Measure the flow rate three times and calculate the mean flow rate valve.

3.4.11 Record the mean flow rate on the SP sheet.

3.4.11.1 If the mean flow rate will give an air volume of 17-23 L during the next collection period, use the same sampler for the collection of the next sample.

3.4.11.2 If the mean flow rate will not give an air volume of 17-23 L during the next collection period, reset the flow to the desired rate and recalibrate the sampler using the procedures described in RTI/ACS-SOP-361-001, Calibration of DuPont P-125A Constant Flow Samplers or use a different sampler which has been set at the proper flow rate.

3.4.12 Enter the pump number, date, time, and new flow rate on the next sample's SP sheet.

3.4.13 Using the unexposed cartridge, repeat steps 3.3.2-3.3.11 for the collection of the next sample.

3.4.14 Return the collected sample to the Tenax storage area.

3.4.14.1 Store the collected sample in a metal can designated for fixed site samples only.

3.4.14.2 Store the collected sample with the other fixed site samples in chronological order of collection until the can contains no more than 17 cartridges.

NOTE: In order to facilitate the final QA check and packing of the cartridges prior to shipment, the collected cartridges should be stored in numbered cans as they are collected. As a result, the cartridges are grouped in order of their collection dates as well as by sets. Once a can is full, the COC sheets may be easily matched with their corresonding samples and grouped together by can number.

3.4.15 Complete the COC sheet for the collected sample including a quality assurance check by another field staff member.

NOTE: A quality assurance check involves a complete check for information contained on the COC sheet including all calculations, signatures, dates, labels and times.

3.4.16 Complete the COC sheet for the sample just exposed.

3.5 Collection of the Final Sample

The collection of the final sample is similar to the collection of any previous sample except the DuPont sampler is collected and recharged.

3.5.1 Transport the following sampling equipment to the sample site:

3.5.1.1 The empty culture tube in which the exposed cartridge will be placed.

3.5.1.2 The bubble flow meter and stopwatch.

3.5.1.3 The forceps and Kimwipes®.

3.5.2 Remove the metal fixed site box from its location.

*3.5.3 Repeat steps 3.4.2-3.4.11 for the collection of the final sample.

NOTE: Pump calibration must be performed within one hour after the sample collection period.

3.5.4 Place the DuPont samples in an area allowing it to discharge.

*3.5.5 Turn off the samplers, place them on the battery chargers at the full charge rate.

NOTE: A charging time of 16 hours at the full charge rate, is recommended, but not mandatory in order to insure a fully charged sample for the next use. Pumps used overnight, replaced in the morning, and needed that afternoon may be charged during the time available.

After 16 hours on full charge the samplers may be removed and stored or switched if trickle charge and left on the chargers.

3.5.6 Return the collected sample to the Tenax storage area. See Sections 3.4.14-3.4.15 for details on storage.

3.5.7 Complete the COC sheet for the collected sample including a quality assurance check by another field staff member.

4.0 LOGISTICAL PROBLEMS

In the course of a field sampling trip, many events may occur which make the collection of fixed site air samples difficult if not impossible.

4.1 Failure to Contact Participant

In the event a participant cannot be contacted for their scheduled appointment and the appointment is cancelled, the fixed site samples must be reassigned. The set of samples should be assigned to the next eligible participant. The next scheduled participant from the same segment should be assigned a fixed site sample.

4.2 No Proper Sampling Site

In some instances, a participant may live in a location unsuitable for the collection of a fixed site air sample. Such cases include high rise apartment buildings with no accessable windows and locations with no secure place to station the sampler. For these cases, reassign the sample set to the next eligible participant. Again, the next scheduled participant in the same segment should be assigned a fixed site sample.

RTI/ACS-SOP-332-001
April 1984
Revision 1

Standard Operating Procedure for the Collection of Water Samples

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Approved:

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RW Handy 4/13/84
QA Officer, RTI/ACS Date

E Pellizzari 4/26/84
Vice-President, RTI/ACS Date

Standard Operating Procedures for the
Collection of Water Samples
RTI/ACS-SOP-332-001

1.0 PRINCIPLE

Water samples are collected at two different times from the source where the participant obtains their drinking water. Since the target compounds in water are volatiles, the samples must be kept refrigerated until analyzed.

2.0 MATERIALS

2.1 Two ounce amber bottles containing sodium thiosulfate
2.2 Foil-lined phenolic resin caps with Teflon® inserts, size 18
2.3 Ice chest and cold packs
2.4 Storage refrigerator

3.0 PROCEDURE

The collection of water samples is basically a simple procedure. Ideally, the collection bottles should be filled so that no headspace is observed once they are capped. With practice, it can be accomplished very easily. Each sample bottle contains a preservative; therefore, it is important not to overfill the bottles and flush out a portion of the preservative. Once collected, the samples must be placed in a cooler with a cold pack until they can be transferred to a storage refrigerator. Ice placed in plastic bags may be substituted for cold packs, if necessary.

3.1 Water Sample Collection Schedule

3.1.1 Refer to the Sample Collection Schedule for water samples and determine what samples are to be collected for the participant. See Figure 1.

NOTE: All samples, field, duplicate, and quality assurance are collected in the same manner.

SITE — (SEASON) — WATER (WV)

	Samples Collected			
Participant Number	Field	Duplicates	QA	Expose QC Set
21451-0	2			
21452-8	2			
21453-6	2	2		
21454-4	2			
21455-1	2			
21456-9	2		2	1
21457-7	2			
21458-5	2			
21459-3	2			
21460-1	2			
21461-9	2			1
21462-7	2			
21463-5	2	2		
21464-3	2		2	1
21465-0	2			
21466-8	2			
21467-6	2			
21468-4	2			
21469-2	2			
21470-0	2			
21471-8	2		2	
21472-6	2			1
21473-4	2	2		
21474-2	2			

Figure 1. Sample Collection Schedule (Example)

3.1.2 Prepare the labels and Chain-of-Custody (COC)/Field Sampling Protocol (SP) sheets for all of the samples to be collected for the participant. See Figures 2 and 3.

3.1.3 Obtain the appropriate number of sample bottles and label each one.

3.1.4 Prepare each label with a ball point pen (black ink only); do not use felt-tip pens.

3.1.5 Affix the label to the jar by encircling the bottle and label with scotch tape.

3.2 Collection of the Water Sample

3.2.1 Transport the sampling bottle, cooler, and ice pack to the participant's residence.

*3.2.2 Ask the participant if they drink tap or bottled water. If they drink tap water, ask the participant from which tap they obtain their drinking water.

NOTE: If bottled water is used by the participant, it should be used when collecting the sample.

3.2.3 If a water purifier is in place, ask the participant if it is used when drawing drinking water from the tap.

3.2.4 If the water purifier is used by the participant, record the information on the SP sheet.

NOTE: If the water purifier is used by the participant, it should be used when collecting the sample.

*3.2.5 If tap water is used, turn on the cold water tap to a moderate rate and allow it to run for 30 seconds.

NOTE: This is necessary in order to purge the water which has been sitting in the lines.

*3.2.6 If tap water is used, slow the water flow and carefully fill the collection bottle.

CAUTION

Do not allow the bottle to overfill or the preservative contained within the bottle may be lost.

*3.2.7 If bottled water is used, allow it to run for 5 seconds before carefully filling the collection bottle.

3.2.8 Carefully cap the bottle and immediately place into the cooler.

*3.2.9 Record the time of collection, date, source of water, and volume collected on the sample SP sheet. A filled, 2 oz bottle contains 64 mL.

FIELD SAMPLING PROTOCOL SHEET - TEAM STUDY

DATE: ______

SAMPLE CODE: INAIR 111-5 WV-F2

AIR SAMPLING

Pump Model: ______ Serial Number: ______

Time (Initial): ______ Flow Rates (Initial): ______

(Final): ______ (Final): ______

Total Time: ______ Avg. Flow Rate: ______

Total Volume Sampled: ______

Temperature (°F): ______

BREATH SAMPLING

Meter Readings Final: ______ ft^3 Time of Day: ______

Initial: ______ ft^3 Temperature: ______ °F

Total: ______ ft^3 Duration of Exhalation __min__sec

Correction Factor: X ______ Pump No.: ______

Volume/Cartridge: ______ L ($1\ ft^3 = 28.3\ L$)

[✓ DRINKING WATER], BLOOD, URINE, MILK SAMPLING

Time of Day: 1900 Source (water only) kitchen tap
(e.g. ground, [✓ city], etc.)

Amount Collected: 60 mL

FOOD SAMPLING

Time Period During Which Food was Consumed: ______

Time of Collection: ______

Container	Container Type	Description of Food Type
(1)		
(2)		
(3)		

GENERAL REMARKS

Figure 2. Field Sampling Protocol Sheet (Front)

CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute, Analytical Sciences Division
Chemistry and Life Sciences Group, Research Triangle Park, NC 27709

SAMPLE CODE INAIR 111-5 WV-F2	**TENAX BATCH:**	**VOLUME COLLECTED:**
SAMPLE TYPE: Water	**TEMPERATURE (°F)**	**VOLUME ANALYZED:**

RELIN-QUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	JS	1900	7/11/83	Sample collected
JS				

Figure 3. Chain-of-Custody Sheet (Back)

3.2.10 Return the samples to the work room and store in a refrigerator.

CAUTION

Do not set the temperature of the refrigerator below 2°C otherwise the samples may freeze and break the sample bottle. Do not store any samples directly beneath the freezer area of the refrigerator since they may freeze.

RTI/ACS-SOP-350-002
December 1983
Revision 0

Standard Operating Procedure for the Maintenance and Operation of the Van

Author:

Jeffrey T. Keever
Jeff Keever

Approved:

L Sheldon
Laboratory Manager, RTI/ACS
12/4/83
Date

RWHandy
QA Officer, RTI/ACS
12/5/83
Date

E Pellizzari
Vice-President, RTI/ACS
12/5/83
Date

Standard Operating Procedure for the Maintenance and Operation of the Van

RTI/ACS-SOP-350-002

1.0 PRINCIPLE

Maintenance records for the operation of the vans used during field sampling operations must be maintained on a regular basis. The records include daily mileage, fuel purchases, daily and weekly maintenance.

2.0 MATERIALS

2.1 Maintenance and Use Form (see Figure 1).

3.0 PROCEDURE

3.1 Place copies of the Maintenance and Use form in all vans used during field sampling operations and travel to and from the sampling site.

3.2 Enter the beginning mileage on the maintenance form regardless of the weekday on which use begins.

3.3 At the end of each day, enter the mileage under the appropriate daily mileage section.

3.4 When purchasing fuel or oil, enter the date, amount purchased, cost, and current mileage under the fuel purchases section of the maintenance form. Retain receipt.

3.5 Indicate any maintenance, adjustments, or additions of fluids required during the daily and weekly maintenance checks.

3.6 Indicate the date on which the van is washed.

3.7 List any comments concerning maintenance, handling, braking, or problems at the bottom of the maintenance form.

3.8 Return all completed forms and receipts to RTI.

MAINTENANCE & USE FORM
(COMPLETE FORM IN DUPLICATE)
LEASE VAN (CCB) LICENSE

BEGINNING MILEAGE ________ FOR THE WEEK ENDING ________

	DATE	ENDING MILEAGE (driver use)	DRIVER	VEHICLE LOCATION	PROJECT CHARGE
Monday					
Tuesday					
Wednesday					
Thursday					
Friday					
Saturday					
Sunday					

TOTAL MILES TRAVELED ________

AVERAGE MILES/DAY ________

FUEL PURCHASES:

Date ________ Gallons ________ /$ ________ Mileage ________

Date ________ Gallons ________ /$ ________ Mileage ________

Date ________ Gallons ________ /$ ________ Mileage ________

Date ________ Gallons ________ /$ ________ Mileage ________

DAILY VEHICLE CHECK:	Oil ☐	Water ☐	Tires ☐	Auto Transmission ☐
Quantity Added				
WEEKLY VEHICLE CHECK:	Battery ☐	Lights ☐	Power Steering ☐	Brake Fluid ☐
Quantity Added				

PERIODIC MAINTENANCE:	Due	Done
Wash (as needed, or 2 times/month		
SCHEDULE "A"	6,000 miles	
SCHEDULE "B"	12,000 miles	
SCHEDULE "C"	18,000 miles	

COMMENTS: (any unusual noises, changes to vehicle handling, brakes, any unsafe condition) ________

Figure 1. Maintenance and Use Form

RTI/ACS-SOP-337-001
April 1984
Revision 1

Standard Operating Procedure for the Collection of Breath Samples

Author:

Jeffrey T. Keever
Jeff Keever

Approved:

L S Sheldon
Laboratory Manager, RTI/ACS — 4/24/84 Date

QA Officer, RTI/ACS — 4/13/84 Date

E Pellizzari
Vice-President, RTI/ACS — 4/26/84 Date

Standard Operating Procedure for the Collection of Breath Samples

RTI/ACS-SOP-337-001

1.0 PRINCIPLE

A breath sample shall be collected for each participant in the study. The sample is collected by using an apparatus which, in its simplest form, is two bags connected by a one-way mouth-piece. One bag, filled with purified and humidified air provides the air supply for the participant to breathe. The other identical bag is used to collect and hold the breath expired by the participant. Once collected, the sample is evacuated from the bag through a Tenax cartridge on which the target compounds are absorbed.

2.0 MATERIALS

2.1 Tedlar bags, 40 Liter; Nutech Inc.
2.2 Compressed air, 0.1 THC grade
2.3 Compressed helium, 99.9% grade
2.4 Distilled water
2.5 Teflon tubing, 1/4" o.d.
2.6 Charcoal filters
2.7 Stainless steel Cajon Ultra Torr Union 316 with viton o-rings
2.8 Stainless steel straight unions, 1/4"
2.9 Teflon ferrules, 1/4"
2.10 Nutech Model 221 Gas Samplers (high volume gas pump)
2.11 Auto batteries, 12 volts
2.12 Drierite, indicating
2.13 Drierite, nonindicating; baked to 400°C for 2 hours
2.14 Glass beaker, 10 mL
2.15 Plywood bag press
2.16 Stopwatch/calculator
2.17 Noseclip

2.18 Mercury thermometers

2.19 Sampling line

- 2.19.1 Latex tubing, 1/4" i.d.
- 2.19.2 Male Quick-Connect, single-end shut off
- 2.19.3 Plastic in-line filter
- 2.19.4 Cajon Ultra Torr Unions 316
- 2.19.5 Stainless steel needles, 1 1/4", 21 ga.
- 2.19.6 Thermogreen septum; Supelco 2-0668
- 2.19.7 Glass pipet, 1/4" i.d. x 3 cm

2.20 Teflon mouthpiece

- 2.20.1 Teflon plug valves, 1/4"; Cole Parmer-6392-20
- 2.20.2 Teflon tubing, 1/4" o.d.
- 2.20.3 Stainless steel ball valve with Teflon seats, 1"
- 2.20.4 Nacom Teflon unions, 1"
- 2.20.5 Viton o-rings

2.21 Humidifier

- 2.21.1 Flat bottom boiling flask, 500 mL, 24/40 joint
- 2.21.2 Midget impinger stopper, 12/5 ball joint, 24/40 stopper Lab Glass-6891
- 2.21.3 Ground socket joint, 12/5 joint; Lab Glass-1041
- 2.21.4 Pinch clamps, size 12; Lab Glass-1045

2.22 Miscellaneous equipment

- 2.22.1 Complete tool set
- 2.22.2 Large forceps
- 2.22.3 Small forceps
- 2.22.4 Empty culture tubes
- 2.22.5 Glass wool
- 2.22.6 Kimwipes®, 8x5 in.
- 2.22.7 Aluminum foil
- 2.22.8 Methanol, glass distilled
- 2.22.9 Small stool
- 2.22.10 Copper wire
- 2.22.11 Three-prong clamps, various sizes

2.22.12 Clamp holders

2.22.13 Small plastic bags

*2.22.14 Spirometer Use Log

3.0 PREPARATION

The following information describes the initial setup and preparation of the sampling equipment used in the collection of breath samples. The equipment should be assembled and tested as soon after arrival at the work site as possible.

3.1 Preparation of the Tedlar Bags

The Tedlar bags used are specifically designed to fit the custom Teflon mouthpiece of the spirometer. Each bag is constructed to hold 40 liters of air. Tedlar is a brittle material and must therefore be handled with care. It should never be folded or creased since this may cause it to crack or split. Inhale and exhale bags, although identical, should always remain segregated in order to minimize cross contamination.

3.1.1 Label each bag either inhale or exhale. One inhale bag for every three exhale bags is the normal ratio.

3.1.2 Attach the in-line charcoal filter to the helium tank.

3.1.3 Fill the bags with helium flowing through the charcoal filter.

3.1.4 Clamp the opening closed with a two-inch binder clip and hang from the grommet.

3.1.5 To purge the bag, fill with helium and set aside the helium-filled bags for at least two hours.

3.1.6 Remove the binder clip, place the bag into the wooden bag press and allow the helium to flow out under the weight of the press.

3.1.7 Repeat steps 3.1.5 and 3.1.6 at least two times.

NOTE: If new Tedlar bags are used, Sections 3.1.5 and 3.1.6 will be repeated at least six times prior to use.

CAUTION

Do not over-inflate the bag. When using the bag press, allow the weight of the press to force the helium from the bag. Excessive pressure applied to the press may damage the bag. Do not fold the mouth of the bag when sealing with the binder clips.

3.2 Preparation of the Nutech 221 Samplers

The Nutech Model 221 Gas Sampler is a high volume gas pump used to draw the collected breath sample from the Tedlar bag through a Tenax cartridge. Two samplers are operated simultaneously, splitting the collected breath sample into two equal portions and drawing it through two parallel Tenax cartridges. Each sampler is powered by a standard 12-volt automotive battery.

3.2.1 Check all tubing connections in the Nutech sampler making sure they are secure.

3.2.2 Install fresh indicating Drierite in the filter, if needed.

3.2.3 Make sure the sampler number and correction factor are displayed on the front panel.

3.2.4 Inspect the DC power cable for damage.

3.2.5 Install two samplers and two batteries in the van.

3.2.6 Attach a sampling line to each Nutech sampler using the male Quick-Connect fitting. See Figure 1 for diagram of sampling line.

3.2.7 Fill the in-line filter of the sampling line with indicating Drierite.

3.2.8 Inspect the 21 ga. needle on the sampling line daily for blockage.

CAUTION

The 21 ga. needle may become blocked after several uses making it impossible to draw the breath sample through the Tenax cartridge.

3.2.9 Replace the needle securing it tightly to the fitting.

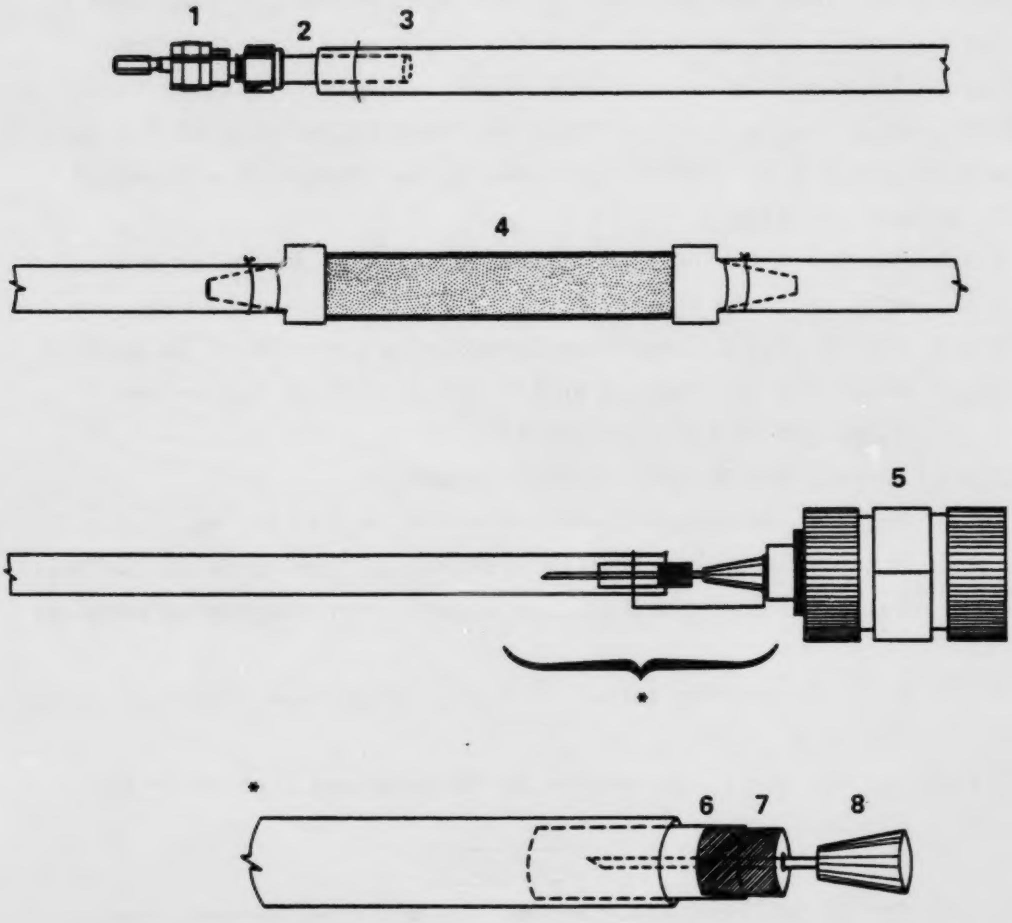
3.2.10 Connect the Nutech samplers to their batteries.

3.2.11 Flip the DC power switches on.

3.2.12 Using the needle valve on the Nutech sampler, adjust the flow so that a back pressure reading between 6 and 7 inches Hg is observed on each sampler.

CAUTION

It is imperative that the pressure drop during sample collection not exceed 7 inches Hg. Possible loss of target compounds will result. A normal sample collection (30 liters of breath divided evenly between two cartridges) should take approximately 15 minutes.



1. Male quick connect
2. Teflon tubing, ¼"
3. Latex tubing, ¼" i.d.
4. Drierite filter
5. Cajon Ultra-torr union
6. Glass pipet, ¼" i.d.
7. Thermogreen septa
8. 1½" Stainless steel needle, 18 ga.

Figure 1. Sampling line and fittings.

3.2.13 Observe the rate at which each sampler is operating for several minutes.

3.2.14 Using the needle valves on the samplers, adjust the flow rates so that they appear to be as equal as possible while maintaining comparatively equal vacuum readings.

3.2.15 Turn the samplers off and disconnect from the batteries.

3.3 Preparation of the Spirometer Mouthpiece

The spirometer mouthpiece is a custom built apparatus constructed of Teflon pipe, a stainless steel ball valve, and two Tedlar flap valves. It allows air to flow in one direction from the inhale bag to the participant, then from the participant to the exhale bag. The design allows the exhale bag containing the breath sample to be completely isolated from the environment after the sample is collected. Individual mouthbits are installed for each participant making it unnecessary to sterilize the entire mouthpiece between uses.

3.3.1 Assemble the mouthpiece, as seen in Figure 2.

3.3.2 Check the two Tedlar flap valves contained in the mouthpiece union by breathing in and out through the mouthpiece. Replace if damaged (i.e., if air flow is obstructed).

CAUTION

The flap valves operate in only one direction. They must be installed so that air will flow in one direction only.

3.3.3 Tighten all connections.

3.3.4 Place two large viton o-rings on both the inhale and exhale sides of the mouthpiece.

NOTE: The o-rings are rolled over the mouth of the Tedlar bags sealing them against the Teflon mouthpiece.

3.4 Assembly of the Spirometer

The entire spirometer apparatus is assembled in the van. It is anchored by the compressed air tank which lies beneath the spirometer body on wooden mounts. See Figure 3 for the approximate orientation of the apparatus in the van. The mouthpiece is mounted on an adjustable bar located on the front of the spirometer body. The glass Y, on which the Tenax cartridges will be attached, is secured to the frame of the spirometer and the floor of the van.

3.4.1 Place the spirometer upright in the van and secure it with the compressed air cylinder.

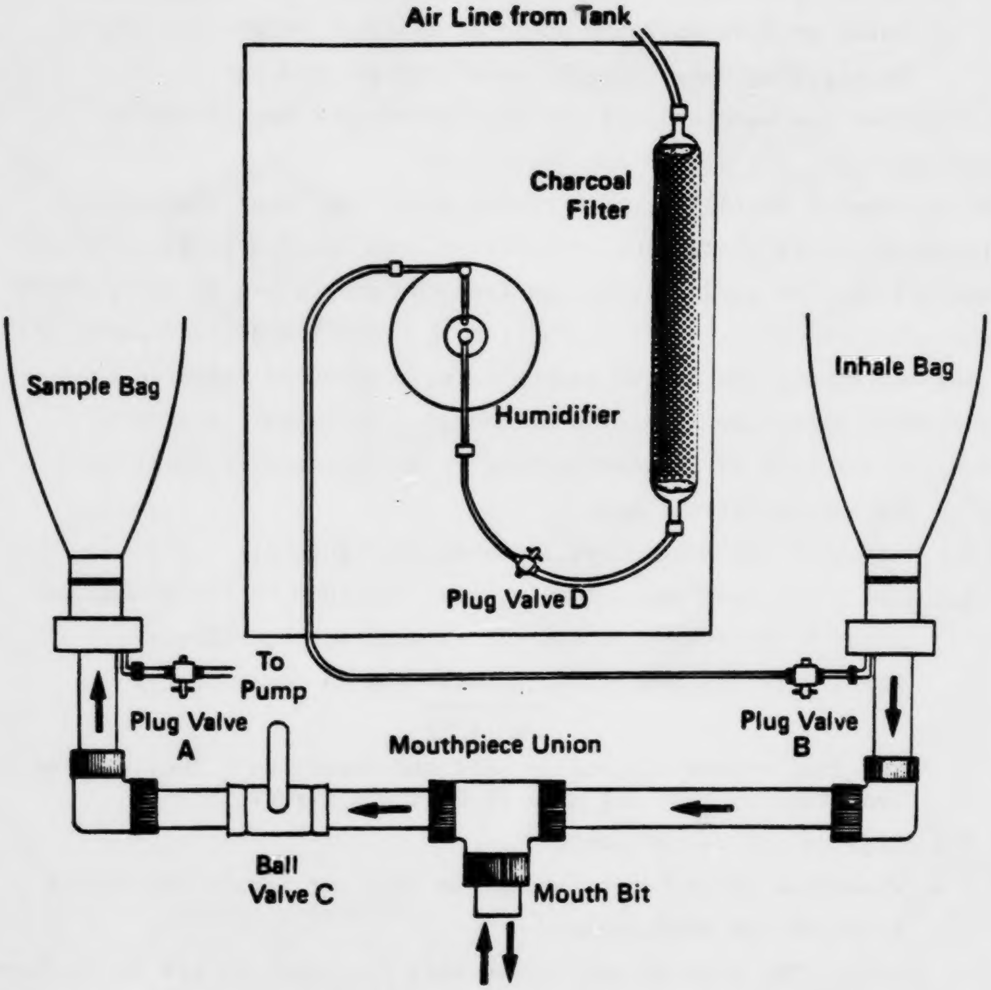

Air Line from Tank
Charcoal Filter
Humidifier
Plug Valve D
Sample Bag
Inhale Bag
To Pump
Plug Valve A
Plug Valve B
Mouthpiece Union
Ball Valve C
Mouth Bit

Figure 2.0. Spirometer apparatus.

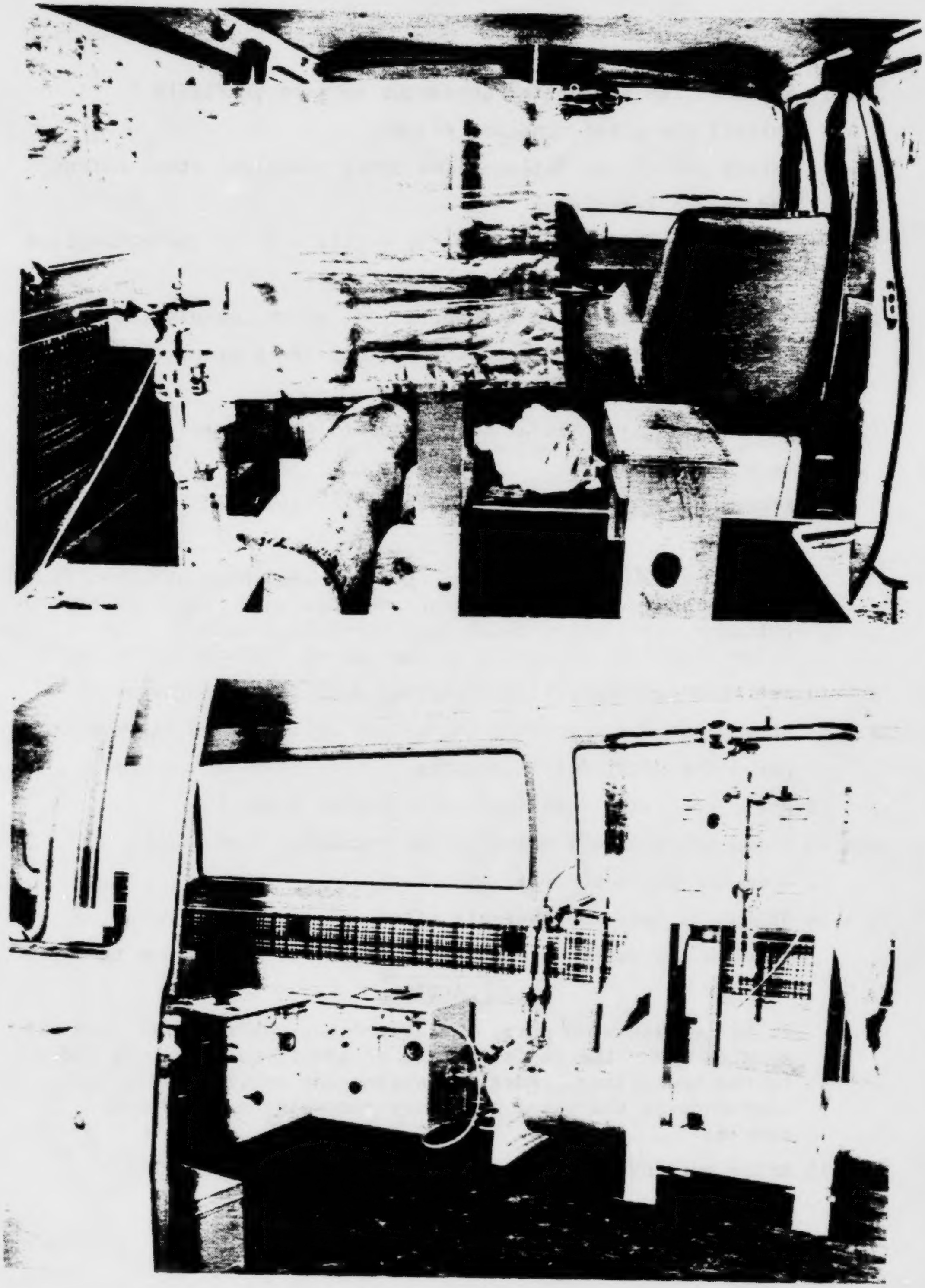


Figure 3. Spirometer in van.

3.4.2 Attach the mouthpiece to the spirometer.

3.4.3 Assemble the humidifier apparatus as seen in Figure 2.

3.4.4 Install the glass charcoal filter.

3.4.5 Attach all of the Teflon lines using stainless steel unions and Teflon ferrules.

3.4.6 Attach the Teflon line from the exhale side of the mouthpiece to the glass Y and secure the fitting.

3.4.7 Secure the glass Y to a support rod, approximately two feet above the gas cylinder, using a small three-prong clamp and copper wire.

3.4.8 Attach two Cajon Ultra Torr unions to the glass Y as depicted in Figure 4.

3.4.9 Attach the gas inlet line to the air regulator.

WARNING

The van should not be driven with the regulator attached to the gas cylinder. Sudden movements may fracture regulator stem or cylinder valve which could lead to serious injury. The cylinder valve should be protected by the cap at all times when not in use.

3.4.10 Fill the humidifier flask halfway with distilled water.

3.4.11 Attach the air regulator to the gas cylinder and vigorously purge the water for 15 minutes.

3.4.12 Refer to Figure 2 and open plug valves B and D.

3.4.13 Close the shut-off valve on the regulator then slowly open the compressed air cylinder.

3.4.14 Gradually open the shut-off valve until a steady bubbling through the water is observed. Adjust the diaphragm to 3 psi.

CAUTION

It is necessary to purge the spirometer water for at least 15 minutes each time it is changed or additional water is added to the humidifier. Purging removes any residual volatile compounds in the water which may contaminate the breath samples.

3.4.15 After purging the water, turn off the air supply at the regulator.

3.4.16 Close plug valves B and D (see Figure 2).

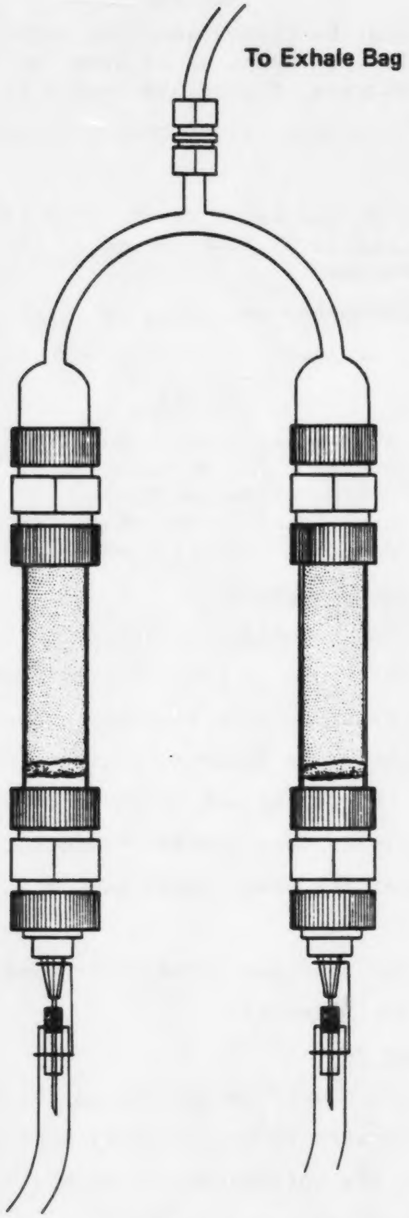

To Exhale Bag
To Nutech Pump

Figure 4. Tenax cartridge and glass Y assembly.

CAUTION

Plug valve D must be closed whenever water is stored in the humidifier. If left open, water from the humidifier will saturate the charcoal filter and render it inactive.

3.4.17 Close the air cylinder valve, remove the regulator, and cap the tank.

NOTE: Freshly purged water may be used for up to one week before being replaced if it is kept sealed in the humidifier and purged each day before use.

3.4.18 Prepare the spirometer mouthbits by rinsing in methanol and air drying for at least 12 hours prior to use.

WARNING

Methanol is flammable and toxic and at no time is to be used or stored in the van. The mouthbits must be cleaned only in a designated area of the workroom. Never allow the mouthbits to soak in methanol longer than five minutes. Air-dry and store in an upright position on a Kimwipe® or clean towel.

3.5 Breath Sample Collection Schedule

3.5.1 Refer to the Sample Collection Schedule (Figure 5) and determine which samples are to be collected from each participant and the Tenax batch numbers for those samples.

3.5.2 Prepare the labels and Chain-of-Custody (COC)/Field Sampling Protocol (SP) sheets for all of the samples to be collected for the participant (see Figures 6 and 7).

3.5.3 Remove and label the appropriate numbers of Tenax cartridges required.

3.5.4 Store all of the labelled Tenax cartridges in the Tenax storage area until needed.

3.6 Supplies Stored in the Van

The following materials should be carried in the van at all times. They include materials necessary in the collection of each breath sample as well as spare parts for the spirometer and Nutech samplers.

3.6.1 Drierite, indicating

3.6.2 Drierite, nonindicating

3.6.3 Mercury thermometer

City ____________ (SEASON) - Breath (BR) ____________

Batch # ____________

Participant Number	Samples Collected Field	Duplicates	QA	Expose QC Sets
21476-7	1			
21477-5	1			
21478-3	1			
21479-1	1		1	
21480-9	1			
21481-7	1			
21482-5	1	1		
21483-3	1			
21484-1	1			
21485-8	1			
21486-6	1		1	
21487-4	1			
21488-2	1			
21489-0	1			
21490-8	1			
21491-6	1			06
21492-4	1	1		
21493-2	1			
21494-0	1		1	
21495-7	1			
21496-5	1			
21497-3	1			
21498-1	1			
21499-9	1			
214500-4	1			

Figure 5. Sample collection schedule for breath (example).

FIELD SAMPLING PROTOCOL SHEET - TEAM STUDY

DATE: ______________________

SAMPLE CODE: ____

AIR SAMPLING

Pump Model: ______________ Serial Number: ______________

Time (Initial): ____________ Flow Rates (Initial): __________

(Final): ____________ (Final): __________

Total Time: ____________ Avg. Flow Rate: ______________

Total Volume Sampled: ______________

Temperature (°F): ________________

BREATH SAMPLING

Meter Readings Final: ________ ft^3 Time of Day: ______________

Initial: ________ ft^3 Temperature: ______________ °F

Total: ________ ft^3 Duration of Exhalation __min__sec

Correction Factor: X __________ Pump No.: ________________

Vol./Cartridge: _____L (1 ft^3=28.3L) Pumping Time: _____ min. _____ sec.

DRINKING WATER, BLOOD, URINE, MILK SAMPLING

Time of Day: ______________ Source (water only) __________
(e.g. ground, city, etc.)

Amount Collected: __________

FOOD SAMPLING

Time Period During Which Food was Consumed: ________________

Time of Collection: __________

Container	Container Type	Description of Food Type
(1)	____________	________________
(2)	____________	________________
(3)	____________	________________

GENERAL REMARKS

Figure 6. Chain-of-Custody Sheet (front)

CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute, Analytical Sciences Division
Chemistry and Life Sciences Group, Research Triangle Park, NC 27709

SAMPLE CODE	TENAX BATCH:	VOLUME COLLECTED:
SAMPLE TYPE:	TEMPERATURE (°F)	VOLUME ANALYZED:

RELINQUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED

Figure 7. Chain-of-Custody Sheet (back)

*3.6.4 Distilled water (approx. 200 mL, for humidifier)
3.6.5 Dummy Tenax cartridge
3.6.6 Kimwipes®
3.6.7 Large forceps
3.6.8 Glass beaker, 10 mL
3.6.9 Spare automotive battery, 12 voltes
3.6.10 Complete humidifier apparatus
3.6.11 Stainless steel needles
3.6.12 Cajon Ultra-Torr Unions
3.6.13 Large viton o-rings
3.6.14 Plywood bag press
3.6.15 Tedlar flap valves
3.6.16 Glass Y's
3.6.17 Spare air regulator
*3.6.18 Spirometer Use Log

4.0 PROCEDURE

The collection of a breath sample is usually the last sample collected from a participant. The sample is collected during the final appointment with the participant. The procedure involves filling a Tedlar bag with purified, humidified air which the participant inhales using a special mouthpiece. The participant then exhales into the mouthpiece filling an empty Tedlar bag with breath. The breath from the filled exhale bag is then drawn through a Tenax catridge (two cartridges in parallel are used for duplicate collections).

4.1 Presampling Preparation

At least 30 minutes prior to leaving the work room for the breath collection appointments, the humidifier water should be purged and the Tedlar bags flushed and filled with fresh helium.

4.1.1 Select one inhale bag for the day and one exhale bag for each scheduled participant.

4.1.1.1 Inspect each bag for small holes or cracks.

4.1.1.2 If the bag has been used frequently and the mouth is very wrinkled or cracked, it may be trimmed back provided it will still fit onto the mouthpiece.

4.1.2 Using the wooden bag press, expel the helium from each bag.

4.1.3 Refill each bag with helium and seal the mouth with a two-inch binder clip.

4.1.4 Obtain a solid Teflon mouthbit (to be used as a plug) and one clean mouthbit for each scheduled participant.

4.1.5 Remove the appropriate Tenax cartridges from the Tenax storage area and place into a field collection can.

4.1.6 Carry the inflated bags to the van and hang on the hooks provided.

CAUTION

Do not allow the bags to fall or lie on the floor of the van. They are easily damaged by grit, sand, or sharp objects.

*4.1.7 Vent the humidifier unit by disconnecting the ball and socket joint between the humidifier and the carbon trap. Immediately reattach the ball and socket joint (see Figure 2).

4.1.8 Attach the air regulator to the air cylinder and purge the humidifier water for approximately five minutes according to Sections 3.4.19 through 3.4.16.

NOTE: This operation may be performed at any time 1 hour prior to filling the first inhale bag with humidified air, except while in transit.

4.2 Equipment Setup

Once the van has arrived at the participant's residence, the equipment should be made ready for the collection of the breath sample. This involves filling the inhale bag with humidified air, checking the Nutech sampler's operation, inserting a dummy Tenax cartridge in-line if needed, and completing the initial paperwork.

4.2.1 Open plug valves B and D (see Figure 2).

4.2.2 Close plug valve A and ball valve C (see Figure 2).

4.2.3 Using the bag press, expel all of the helium from the inhale bag.

4.2.4 Immediately install the inhale bag on the right side of the spirometer mouthpiece.

4.2.5 Hang the bag by the grommet from the hook on the spirometer body.

4.2.6 Carefully roll the two large o-rings over the mouth of the bag, thus sealing the bag against the Teflon mouthpiece.

4.2.7 Place the solid Teflon mouthbit into the mouthpiece.

4.2.8 Attach the air regulator to the air cylinder and carefully bleed air through the humidifier into the inhale bag.

CAUTION

Never open the air cylinder before closing the shutoff valve on the regulator. A sudden surge of air may destroy the humidifier. Keep the back pressure relatively low, controlling the air flow with the shutoff valve.

4.2.9 Fill the inhale bag with air, but do not over-inflate.

4.2.10 While the bag is being filled, connect the Nutech samplers to the batteries and determine if they are both operational.

*4.2.11 Check the critical orifice needles on both sampling lines for blockage by turning on both Nutech pumps for a short time (30 sec). A plugged orifice will cause the back pressure to increase sharply.

*4.2.12 If only one sample is to be collected, i.e., one Tenax cartridge, insert the dummy Tenax cartridge into the Cajon fitting on one of the sampling lines and carefully insert into the Cajon fitting on one arm of the glass Y. See Figure 4 for the proper orientation of the Tenax cartridge.

NOTE: (1) A standard breath sample constitutes only one Tenax cartridge; therefore, an unused or dummy Tenax cartridge is placed in line with the second Nutech sampler. If duplicate samples are to be collected for a participant, a clean Tenax cartridge is used in place of the dummy Tenax cartridge. (2) If a dummy Tenax cartridge is used, place a distinctive marking on it so that it cannot be mistaken for the actual breath sample.

4.2.13 Insert the void end of the Tenax cartridge into the sampling line fitting, not into the glass Y fitting.

WARNING

Use extreme caution when inserting the Tenax cartridge into the fitting on the glass Y. Excessive force may break the glass Y resulting in personal injury.

4.2.14 Record the Nutech sampler serial number and correction factor on the COC sheet for the sample.

4.2.15 Record the initial dry gas meter reading from the Nutech sampler on the COC sheet.

NOTE: Be sure to record the serial number, correction factor, and meter reading for the sampler which will be used to collect the sample, not the one attached to the dummy cartridge.

4.2.16 Remove the mercury thermometer from its case and place in a safe location near the spirometer.

4.2.17 Ready the stopwatch or timer.

4.2.18 Once the inhale bag is full, turn off the air supply and seal the bag.

4.2.19 Turn off the air cylinder valve and the air regulator shutoff valve.

4.2.20 Close plug valve B (see Figure 2).

CAUTION

Do not close plug valve B before turning off the air supply. The in-line back pressure may force the stopper from the humidifier, contaminating the air within, and possibly destroying the humidifier.

*4.2.21 Record the date, participant number, collector's I.D. and whether the humidifier water or carbon trap were changed on the Spirometer Use Log (Figure 8).

4.3 Collection of the Breath Sample

When collecting the breath sample, it is important to note several things. First, the participant must both inhale and exhale through the mouthpiece while never removing their lips from the mouthbit. They must always wear a noseclip to prevent breathing outside air. They must be cautioned to breath at a normal rate, not quickly, otherwise they may hyperventilate. Most participants breath much too quickly and often require a reminder to slow down. Finally, the participant should fill the exhale bag with their breath without any interruptions. Once they remove their lips from the mouthpiece they may inhale outside air which will contaminate the breath sample if they start breathing into the spirometer once again.

4.3.1 Just prior to the participant entering the van, place the exhale bag on the left side of the spriometer by repeating steps 4.2.2 through 4.2.3.2 using an exhale bag.

SPIROMETER USE LOG

Spirometer I.D.: ____________

Date	Participant Number	Collector's I.D.	Change Humidifier H_2O	Change C-trap

Figure 8. Spirometer use Log.

4.3.2 Remove the participant's Tenax cartridge from the field collection can and install it on the sampling line and glass Y.

NOTE: The Tenax cartridge should be installed only after the participant arrives. This reduces the time which the cartridge is exposed to the ambient air.

4.3.3 Using forceps, remove the top pad of glass wool from the culture tube and lay it on a clean Kimwipe®.

4.3.4 Using a clean Kimwipe®, remove the Tenax cartridge and install the void end into the Cajon fitting of the sampling line (see Figure 4).

4.3.5 Carefully install the other end of the Tenax cartridge into the fitting on the glass Y. See WARNING in Section 4.2.9.

4.3.6 Check all fittings to insure that the Tenax cartridges are securely held in place and sealed with the viton o-rings of the Cajon fittings.

4.3.7 Invite the participant into the van and ask him/her to sit facing the spirometer.

4.3.8 Briefly explain the procedure used and the spirometer function to the participant.

4.3.9 Adjust the mouthpiece to a comfortable height for the participant.

4.3.10 Instruct the participant to breath only through their mouth, to keep their lips sealed around the mouthbit, and not to stop before the exhale bag is full.

4.3.11 Have the participant put on the noseclip so that no ambient air can be inhaled through the nose.

4.3.12 Remove the solid Teflon plug from the mouthpiece and install a clean mouthbit.

4.3.13 Begin the breath collection by having the participant exhale and then place their lips on the mouthbit.

4.3.14 Open ball valve C (see Figure 2).

4.3.15 Instruct the participant to begin breathing.

4.3.16 Start timer to determine duration of breath collection.

NOTE: Having the participant exhale just prior to breathing from the spirometer will remove most of the unclean ambient air from their lungs, therefore, reducing contamination of the breath sample from ambient air.

4.3.17 Observe the participant for a moment and remind them to breathe at a normal rate and to maintain a good seal on the mouthbit with their lips.

4.3.18 Using large forceps remove the bottom pad of glass wool from the culture tube placing it on a clean Kimwipe®.

4.3.19 Fill the 10 mL beaker to the 5 mL mark with the Drierite.

4.3.20 Pour the Drierite into the culture tube and replace the bottom pad of glass wool.

4.3.21 When the exhale bag is full, tell the participant to stop breathing and seal the bag.

4.3.22 Tell the participant to resume normal breathing and to remove the noseclip.

4.3.23 Stop timer and record duration of exhalation on the SP.

4.3.24 Close ball valve C (see Figure 2).

4.3.25 Remove the mouthbit and place the solid Teflon plug into the mouthpiece.

4.3.26 Open plug valve A (see Figure 2).

4.3.27 Record the time of day, date, and temperature on the SP sheet.

4.3.28 Turn on both Nutech samplers and begin pumping the breath sample through each Tenax cartridge.

NOTE: The optimum volume per cartridge is 15 liters of air which corresponds to a total collection of 30 liters. This may mean not completely emptying the exhale bag if the total sample volume is in excess of 30 liters.

4.3.29 When a total of 30 liters have been pumped, turn off both Nutech samplers (15 L through each cartridge) and record the pumping time.

4.3.30 Close plug valve A (see Figure 2).

4.3.31 Using a clean Kimwipe®, carefully remove the Tenax cartridge from the Cajon fittings and place it into the culture tube.

4.3.32 Using forceps place the top pad of glass wool onto the Tenax cartridge and cap the culture tube.

CAUTION

Do not allow excess glass wool to lie over the top edge of the culture tube. This may prevent the cap and septum from forming a proper seal with the culture tube, possibly resulting in the contamination of the sample during storage.

4.3.33 Return the culture tube to the field collection can and seal the can.

4.3.34 Record the final dry gas meter reading from the Nutech sampler on the SP sheet.

4.3.35 Remove the exhale bag from the spirometer, place the bag in the wooden bag press, and allow any remaining breath sample to flow out under the weight of the press.

CAUTION

Do not fold the bag in an effort to force out the air. This may break or damage the bag.

4.3.36 Hang the empty exhale bag on the hooks in the van.

4.3.37 If additional breath samples are to be collected, leave the inhale bag sealed and in place.

4.3.38 If no additional samples are to be collected, remove the inhale bag and secure the spirometer.

4.3.39 Hang the inhale bag from the hooks provided.

4.3.40 Close plug valve D (see Figure 2).

4.3.41 Remove the regulator and cap the air cylinder.

4.3.42 Disconnect the Nutech samplers from their batteries.

4.3.43 Return the Tenax cartridge to the Tenax storage area.

NOTE: Store the collected Tenax in cans by matrix and chronological order.

4.3.44 Fill the used exhale and inhale bags with helium and hang in the work room storage area.

4.3.45 Rinse all used Teflon mouthbits with methanol (see Section 3.4.18).

4.3.46 Complete the sample COC sheets including a quality assurance check by another field staff member.

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December 1983
Revision 0

Standard Operating Procedure for the Shipment of Field Sampling Equipment

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Approved:

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Standard Operating Procedure for the Shipment
# of Field Sampling Equipment
RTI/ACS-SOP-340-001

1.0 PURPOSE

The equipment needed for the collection of field samples shall be transported to the field site in vans. Alternatively, air freight and passenger air flights may be used. The equipment and supplies should be packed to insure their security and integrity during the trip.

2.0 PREPARATION

All equipment must be securely packed before leaving RTI. Fragile equipment should be packed in footlockers and insulated with foam rubber. Solvents should be packed in appropriate cartons and carefully loaded into the vans.

2.1 Spirometer

All glassware from the interior of the spirometer and the Teflon mouthpiece should be removed from the spirometer body and individually packed into footlockers. The top door of the spirometer body should be taped into place.

2.2 Tedlar Bags

The Tedlar breath bags and the Tenax storage bags must be carefully packed. They should be rolled and placed into boxes without folding. No heavy or sharp objects should be placed in the same boxes as the Tedlar bags.

2.3 Packing the Van

The equipment should be packed securely in the vans to prevent any shifting or sliding during the trip. The refrigerator should be packed in an upright position and placed so that it will not overturn if bounced around. The glass shelves and door to the refrigerator should be taped securely in place before packing.

3.0 INVENTORY CHECKLISTS

The following checklists should be used as a guide when collecting and packing the equipment for a sampling trip. Although not specific with regard to exact numbers and details, the lists outline the items necessary for the collection of breath, water, personal air, and fixed site air samples.

3.1 General Supplies

- 3.1.1 Kimwipes®, 8 x 5"
- 3.1.2 Forceps, large and small
- 3.1.3 Plastic bags, small
- 3.1.4 Bottles, 1 qt. ointment
- 3.1.5 Rubber gloves
- 3.1.6 Flashlights
- 3.1.7 Glass wool
- 3.1.8 Cotton
- 3.1.9 Empty paint cans
- 3.1.10 Clamps
- 3.1.11 Clamp holders
- 3.1.12 Calculator/timer
- 3.1.13 Calculator batteries
- 3.1.14 Rubber stamps and pads
- 3.1.15 Tools
- 3.1.15.1 Wrench, 1/4"
- 3.1.15.2 Adjustable wrench for regulators
- 3.1.15.3 Screwdrivers
- 3.1.15.4 Allen wrenches
- 3.1.15.5 Pliers

3.2 Office Supplies

- 3.2.1 Three ring notebooks and dividers
- 3.2.2 Strapping tape
- 3.2.3 Scotch tape
- 3.2.4 Shipping labels

3.2.5 Miscellaneous paper pads
3.2.6 Chain-of-Custody sheets
3.2.7 Preprinted labels
3.2.8 Ink pens; black only
3.2.9 Manila folders
3.2.10 Hole punch
3.2.11 Maps
3.2.12 SOC questionnaires and forms
3.2.13 Binder clips, various sizes
3.2.14 Sample Collection schedules
3.2.15 Paper clips
3.2.16 Stapler
3.2.17 Magic markers
3.2.18 RTI telephone directory

3.3 Water Samples

3.3.1 2-oz. Amber bottles with caps
3.3.2 Ice chests, small
3.3.3 Coolers, large 8 gallon
3.3.4 Ice packs
3.3.5 Refrigerator
3.3.6 Bubble wrap

3.4 Air Samples

3.4.1 DuPont P-125A Constant Flow Samplers
3.4.2 Sampler chargers
3.4.3 Hex keys
3.4.4 Spare parts kit
3.4.5 Air sampling train components
3.4.5.1 Tygon tubing, 1/8" o.d.
3.4.5.2 Tygon tubing, 1/4" o.d.
3.4.5.3 Beckman 518 fittings, top and bottom
3.4.5.4 Gelman glass fiber filters
3.4.5.5 Springs, 2"
3.4.5.6 Plastic culture tubes, modified

3.4.5.7 Alligator clips, small
3.4.6 Bubble flow meters
3.4.7 Ring stands
3.4.8 Snoop
3.4.9 Rubber pipet bulbs, 2 mL
3.4.10 Self adhesive labels
3.4.11 Metal fixed site boxes
3.4.12 Sampling vests, adult and child sizes
3.4.13 Temperature conversion charts, °C to °F
3.4.14 Safety pins

3.5 Breath Samples

3.5.1 Spirometer bodies
3.5.2 Spirometer mouthpieces
3.5.2.1 Extra flap valves
3.5.2.2 Mouthbits
3.5.2.3 Viton o-rings
3.5.2.4 Rubber bands
3.5.3 Teflon tubing, 1/4" o.d.
3.5.4 Flat bottom boiling flasks, 500 mL
3.5.5 Impingers, bent at 90°
3.5.6 Midget impinger stoppers
3.5.7 Pinch clamps
3.5.8 Glass charcoal filters, 1/4" tube fittings
3.5.9 Stainless steel straight unions, 1/4"
3.5.10 Teflon ferrules/back ferrules
3.5.11 Teflon plug valves, 1/4"
3.5.12 Teflon tape
3.5.13 Cajon Ultra Toor Unions, #316
3.5.14 Viton o-rings for Cajon fittings
3.5.15 Glass Y's, 1/4" o.d. inlet; 6mm outlets
3.5.16 Breath sampling line components
3.5.16.1 Stainless steel needles, 18 ga, 1 1/2"
3.5.16.2 Thermogreen septa

3.5.16.3 Latex tubing, 1/4" i.d., thick wall
3.5.16.4 Male Quick-Connects, single end shut-off
3.5.16.5 Indicating drierite
3.5.17 Nonindicating drierite, baked to 400°C
3.5.18 Distilled water
3.5.19 Air regulators, two stage
3.5.20 Helium regulators, two stage
3.5.21 Charcoal in-line filters, 1/4" fittings
3.5.22 Teflon tubing, 1/4"
3.5.23 12 volt automotive batteries
3.5.24 Nutech 221 Constant Flow Sampler unit
3.5.24.1 DC power cables
3.5.24.2 AC power cables
3.5.24.3 Extension cords
3.5.24.4 Nutech 221 Constant Flow Sampler
3.5.25 Power strips
3.5.26 Stools
3.5.27 Tedlar breath bags
3.5.28 Bag supports with hooks
3.5.29 Glass beakers, 10 mL
3.5.30 Methanol, pesticide grade
3.5.31 Mercury thermometers
3.5.32 Noseclips
3.5.33 Plywood bag presses
3.5.34 Tenax storage bag assembly
3.5.34.1 Male Quick-Connects, double end shut-off
3.5.34.2 Female Quick-Connects, bulkhead fitting
3.5.34.3 Wooden dowels
3.5.34.4 Wooden bag clamps
3.5.34.5 Binder clips, 2"
3.5.34.6 Tenax storage bags

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Revision

STANDARD OPERATING PROCEDURES FOR SITE WORKROOM PROCEDURES AND RULES

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STANDARD OPERATING PROCEDURE FOR THE SITE WORKROOM PROCEDURES AND RULES

RTI/ACS-SOP-350-001

1.0 SUMMARY

The site workroom is the area in which all sampling operations are based. Most sampling equipment is stored and prepared in the workroom. All samples are stored in the workroom; therefore, every effort must be made to keep the room free of contamination. Within the workroom, specific equipment and paperwork areas must be assigned and properly maintained to ensure smooth operation and proper account of samples and forms. The Site Administrator is responsible for the workroom and has the authority to enforce all rules and regulations.

2.0 WORKROOM SETUP

Upon first arrival at the workroom, the Site Administrator will be in charge of equipment setup. All site personnel will assist in the setup and familiarize themselves with the location of all equipment and paperwork.

2.1 Furniture

2.1.1 Immediately upon arrival, inspect the workroom to determine what, if any, extra furniture will be needed.

NOTE: Generally, a workroom will need the following: three or four long tables, three lamps, several chairs, a telephone, at least one refrigerator, and at least five, 110 V power outlets.

2.1.2 Request any needed furniture from the hotel staff as soon as possible. In the event the necessary furniture is not available at the hotel, rent the furniture.

2.1.3 Arrange the furniture as needed for power requirements and organization.

2.2 Arrangement of Equipment

2.2.1 Refrigerators

2.2.1.1 Connect refrigerator(s) to power outlet.

2.2.1.2 Place a thermometer on the middle shelf of the refrigerator to be used for storage of water samples.

2.2.1.3 Check the temperature after 4 hours. Adjust the temperature to 4°C ± 2°C.

CAUTION

If the refrigerator temperature reaches 0°C or below water samples may freeze and the sample bottles may break.

2.2.2 Pump-Charging Area

Using as many power strips as needed, set up a pump-charging area for personal sampling pumps.

2.2.2.1 Begin charging the personal sampling pumps.

2.2.2.2 Make arrangements for differentiating between charged and discharged pumps. Label each group of pumps by time and date. Store charged and discharged pumps in different areas of the workroom.

2.2.3 Set up the Tenax GC storage bag and begin purging with helium. Purge with helium for 30 minutes at 100 mL/min. Place all Tenax GC containers in the storage bag and continue purging.

2.2.4 Prepare an area in which the flow rates of personal sampling pumps are measured. Keep a bubble meter, stopwatch, pen, paper, and hex key in the area. Provide light to make accurate flow rate measurements.

2.2.5 Prepare an area, preferrably in a bathroom, for cleaning spirometer mouthpieces. Use an airtight container for storage of methanol.

2.2.6 Store methanol in this container at all times except during actual cleaning of mouthpieces. Also store long forceps, Kimwipes®, and a 1-quart wide-mouth screw-cap jar in this area.

WARNING

Methanol is flammable and toxic. The storage container must be labelled: "METHANOL, FLAMMABLE AND POISONOUS." Every effort must be made to keep the methanol separated from the spirometer water. All containers must be clearly and boldly labelled. Methanol must not be placed in any van for any reason.

2.2.7 Set aside an area for hanging Tedlar spirometer bags. A coat rack, shower curtain rod, or a string hung across a portion of the room is suitable for this purpose. Fill the bags with helium as soon as possible and hang them in the designated area. Hang bags only where they will not be damaged by doors or passing people.

2.2.8 Store spare parts, such as extra sampling trains, gas regulators, fittings, etc., in labelled boxes in a designated area.

2.2.9 Set up a table, preferrably in the center of the workroom, upon which equipment can be prepared and/or paperwork done. The table will be a general work station for all site personnel and will not be used for permanent equipment storage.

2.2.10 Store all containers, boxes, and footlockers not in use where they will not interfere with normal work. Place all miscellaneous equipment in convenient areas of the room.

2.2.11 Upon receipt, store gas tanks in the workroom except for air tanks in use in the vans.

WARNING

All gas cylinders used are under high pressure. Tanks must be stored horizontally on the workroom floor with regulators removed to prevent possible accidents and bodily injury.

2.3 Arrangement of Paperwork

2.3.1 Attach sample collection schedules for personal air, fixed-site air, water, and breath samples to the workroom wall for easy reference. If more than one city is being sampled, separate and clearly label the collection schedules.

2.3.2 Post Tenax GC batch numbers on the workroom wall near the Tenax GC storage area. The batch numbers should correspond to certain sample types and be so labelled.

2.3.3 Label the looseleaf notebooks which contain finished sample protocol sheets by sample matrix. Prepare separate notebooks for each city being sampled. File only those protocol sheets which have had quality assurance checks.

2.3.4 File appointment control forms and household screening forms in clearly labelled folders near the workroom telephone.

2.3.5 Maintain a file for all receipts collected in the course of the sampling trip. The receipts will include, but are not limited to, gas cylinders, sample shipments, rentals, and any miscellaneous expenses.

NOTE: Upon taking receipt for any service or supply, the site personnel will check very closely for accuracy and proper documentation.

2.3.6 Store all spare forms including sample protocol sheets, survey instruments, shipping labels, etc., in a designated, labelled area.

3.0 CONTROL OF CONTAMINATION

Site personnel shall make every effort to avoid contamination of samples, controls, and blanks in the site workroom. Special attention must be given to keeping airborne contaminants away from Tenax GC cartridges since many compounds will adsorb onto the Tenax GC.

3.1 Rules for Site Personnel

3.1.1 Do not bring any cleaning solution other than methanol into the site workroom. Keep the methanol in a tightly capped container when not in use.

3.1.2 Do not use any aerosol spray or insecticide/pesticide in the site workroom.

3.1.3 Keep doors and windows in the workroom closed as much as possible.

3.1.4 Bring any source of contamination to the attention of the Site Administrator. If possible, eliminate the source of the contamination.

CAUTION

To eliminate possible contamination, two general rules must be observed: (1) no smoking of any kind and (2) no cooking allowed in the site workroom.

3.2 Rules for Hotel Staff

The guidelines listed below pertain to nonRTI individuals and, therefore, are expressed as objectives rather than requirements. Although the action of hotel employees and contractors may be outside the control of RTI site personnel, all hotel activities which may have an effect on data quality will be documented.

The RTI staff member who initially contacts the hotel to make room reservations will be responsible for explaining to the hotel management the need for special precautions against contamination during the sample collection and the importance of observing the following rules.

3.2.1 The site personnel shall discuss with the hotel cleaning staff the need to keep all cleaning solutions out of the workroom.

3.2.2 Members of the hotel cleaning staff should enter the workroom only when site personnel are present and perform only the work requested by site personnel.

3.2.3 Hotel maintenance workers should not be allowed to do any painting, wallpapering, glueing, etc., in the workroom. If the maintenance worker insists, the problem should be brought before the hotel manager by the Site Administrator. Should the problem not be resolved the Site Administrator should contact the Project Manager at RTI.

4.0 USE OF REFRIGERATORS

One refrigerator in the site workroom is reserved exclusively for water samples, controls, and blanks. Nothing else may be stored in this refrigerator except ice packs used for sample shipment. The only exception to this rule is if other types of biological or environmental samples, such as urine or food, are collected and need to be kept cold. The refrigerator which should be used for storage of water samples is the compact Kenmore model, or equivalent.

4.1 Do not store water samples on the shelf directly below the freezer compartment because the water may freeze and break the sample container.

4.2 Keep unused water controls and blanks separate from samples so they may be easily identified.

4.3 Monitor the refrigerator temperature (particularly at first) and adjust to 4°C ± 2°C. The temperature should be monitored daily.

4.4 If possible, obtain a second refrigerator with a freezer compartment. The extra freezer space is needed for freezing cold packs used for shipping water samples. The second refrigerator is also used for storing food and drinks for the site personnel. No water samples should be stored in this refrigerator.

5.0 USE OF TELEPHONE

5.1 Telephone Charges

5.1.1 Use the telephone in the site workroom only for business calls.

5.1.2 Charge all long distance calls to the credit card number. The credit card will be posted near the phone.

5.1.3 Do not charge calls to the RTI toll free (800) number.

5.1.4 Make all personal calls from the individual's personal room. No personal calls may be charged to the credit card number.

5.2 Post a list of important telephone numbers next to the workroom telephone. The numbers considered to be important and possible reasons why those people may need to be contacted are presented in the following paragraphs.

5.2.1 List the business and home telephone numbers of the RTI Project Manager. The manager should be contacted during business hours concerning all sample acquisition and planning problems. The manager will be contacted at any time if site personnel or respondents are injured, if a serious auto accident occurs, or if any other major emergency occurs.

5.2.2 List the business and home telephone numbers of the RTI survey specialist. The survey specialist should be contacted in case of any major difficulty concerning appointment scheduling. Also, any questions which a respondent poses that cannot be answered by site personnel should be referred to the survey specialist.

5.2.3 List the business telephone numbers of local town officials and health officers involved in the survey. When respondents have questions concerning survey results or health effects of the target compounds, they may be referred to local health officials.

5.2.4 List the telphone numbers and times of availability for the local interviewers. Many scheduling problems may be solved by contacting the interviewers directly.

5.2.5 Keep a current RTI staff telephone directory near the telephone. During the course of a trip, it may be necessary to discuss logistical problems, equipment and sample shipments, and progress reports with several members of the staff at RTI.

5.3 Telephone Duty

Every day, during a designated time period, one of the site personnel must be in the workroom to accept incoming telephone calls. During this period, interviewers have been instructed to communicate new respondent appointments. Also, the staff at RTI will be assured of finding someone in the workroom during this time period.

The Site Administrator has the responsibility of assigning site personnel to telephone duty on a rotating schedule.

6.0 BILLING AND CHECK-OUT

6.1 Billing Maintenance

The Site Administrator shall have the responsibility of checking and maintaining the site workroom bill.

Unless arrangements are made to directly bill RTI, the workroom charges will be paid by the Site Administrator. The Site Administrator must have sufficient funds to cover all charges.

6.1.1 During extended site visits, thoroughly examine the bill for the workroom approximately once a week. The Site Administrator shall determine that all charges are proper and up to date.

6.1.2 Check all bills and invoices for accuracy and retain all receipts. Return all documentation to RTI and submit with expense report.

6.2 Cleanup and Checkout

The Site Administrator present at the end of sample collection shall be in charge of cleaning the workroom, packing the vans, and checking out of the hotel. All site personnel will assist with the cleaning and loading.

The Site Administrator will checkout of the workroom, making sure that the final billing is accurate. The Site Administrator will also ascertain that all keys used by RTI staff have been returned. It is the responsibility of each individual to settle personal room accounts and checkout.

6.2.1 Securely pack and ship all samples to RTI.

6.2.2 Check all sample survey instruments for completeness and pack into a labelled container.

6.2.3 Collect weather data from the closest source, usually an airport or radio station. Data collected should include hourly temperatures, wind speed and direction, and daily rainfall. Weather data should be filed with the other important paperwork and packed for return to RTI.

6.2.4 Securely pack, dismantle if necessary, and load sampling equipment into the vans for return to RTI. All small parts should be placed in labelled boxes. The vans should be loaded so that equipment is completely immobilized.

6.2.5 Return any rented items such as refrigerators or vans.

6.2.6 Close out the hotel safe deposit box if one was used for securing funds. Any money remaining should be immediately counted and returned to RTI by the Site Administrator.

6.2.7 Inspect the workroom just prior to check out to ensure that no equipment remains. The workroom should be straightened up by site personnel before departing.

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Standard Operating Procedure for the Calibration of DuPont P-125A Constant Flow Samplers

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Standard Operating Procedure for the Calibration of DuPont P-125A Constant Flow Samplers

RTI/ACS-SOP-361-001

1.0 PRINCIPLE

The DuPont sampling pumps used in the collection of air samples must be calibrated before use. The pump batteries must be fully charged and the flow rates adjusted to 25-30 mL/min.

2.0 MATERIALS

2.1 DuPont P-125A Constant Flow Sampler
2.2 Bubble flow meter, 25 mL
2.3 Stopwatch/calculator
2.4 Small flat screwdriver
2.5 Allen Keys (5/64")
2.6 110 volt, 60 Hz battery charger
2.7 Tygon tubing (1/8" i.d.)
2.8 DuPont P-125A Operations Manual

3.0 PROCEDURE

3.1 Using a 5/64" allen key, remove the back of the sampler.

3.2 Flip the on/off switch to the "on" position. It is normal for the sampler to take several seconds to start.

3.3 Check the "battery check" light to determine if the sampler is fully charged (see Figure 1). If the light is "off," place the sampler on a battery charger at full charge for 16 hours or until fully charged.

NOTE: A fully charged battery is necessary if the sampler is to be used in the field.

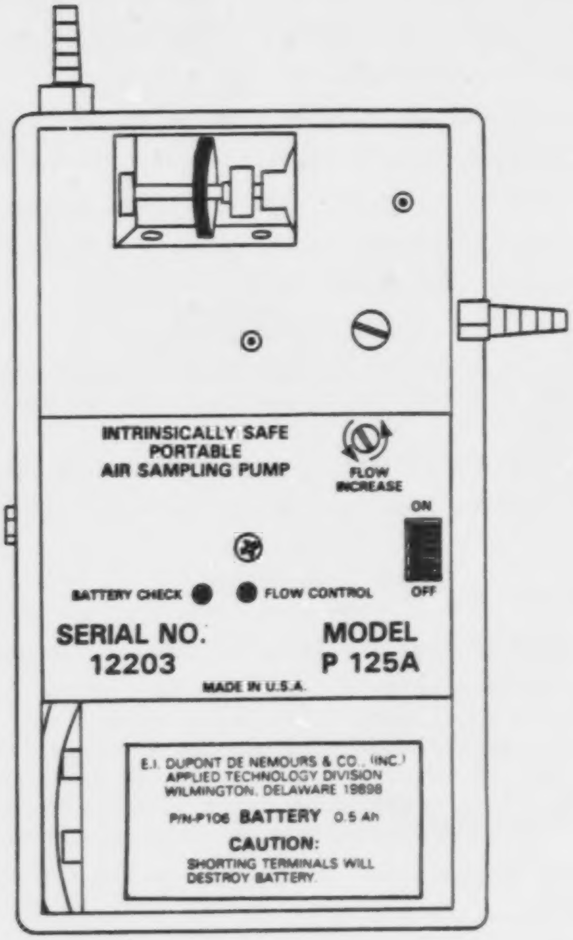
INTRINSICALLY SAFE
PORTABLE
AIR SAMPLING PUMP
FLOW
INCREASE
ON
OFF
BATTERY CHECK
FLOW CONTROL
SERIAL NO.
12203
MODEL
P 125A
MADE IN U.S.A.
E.I. DUPONT DE NEMOURS & CO., (INC.)
APPLIED TECHNOLOGY DIVISION
WILMINGTON, DELAWARE 19898
P/N-P106 BATTERY 0.5 Ah
CAUTION:
SHORTING TERMINALS WILL
DESTROY BATTERY.

Figure 1. DuPont P125-A Constant Flow Sampler (Interior View)

3.4 Determine whether the flow controller is compensating for changes in flow by placing your finger across the inlet barb. If the pump speed increases the controller is functioning properly; if the speed does not increase, check the sampler with a bubble flow meter in the normal manner to verify that the unit is operating at a pumping rate greater than 25 mL/min.

3.5 Allow approximately two minutes for the sampler flow to stabilize.

3.5.1 If the flow control light goes out 15 to 45 seconds after the sampler is started, either the flow adjustment is open too far or the pump lacks capacity. Close the flow adjustment screw and repeat steps 3.2-3.5.

CAUTION

Do not tighten the flow adjustment screw too far in the clockwise direction or damage to the needle valve will result.

3.6 Attach the bubble flow meter to the outlet flow barb of the sampler.

3.7 Measure the sampler flow once using the bubble flow meter and a stopwatch.

3.7.1 $\text{Flow rate} = \dfrac{\text{Bubble tube volume (mL)}}{\text{Elapsed Time (sec.)}} \times \dfrac{60\ \text{sec.}}{1\ \text{min.}} = \dfrac{\text{mL}}{\text{min}}$

3.8 Adjust the flow if the rate falls outside of the desired range.

NOTE: The DuPont sampler fails to perform adequately at flow rates less than 25 mL/min. Flow rates greater than 30 mL/min may result in the collection of samples exceeding the breakthrough volumes of certain compounds on the Tenax cartridge. However, the use of higher flow rates (30-60 mL/min) may be necessary if the sampling period has been shortened.

3.9 Once the flow has been set, attach the back cover and tighten the screw using the allen key.

3.10 Allow at least five minutes for the sampler flow to stabilize.

NOTE: The flow rate is affected by the tightening of the cover screw. Final flow measurements should not be taken for at least five minutes after the back cover is tightened into place.

3.11 Measure the flow rate a total of three times using the method described in Section 3.7.

3.12 Determine the final flow rate by calculating the mean value for the three flow measurements.

    3.12.1 If the mean flow rate is within the desired range record the mean flow rate on the sampler and the appropriate forms.

    3.12.2 If the mean flow rate falls outside of the desired range, steps 3.1 through 3.12 must be repeated.

RTI/ACS-SOP-367-001
December 1983
Revision 0

Standard Operating Procedure for the Calibration of Nutech Model 221 Gas Sampler

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Standard Operating Procedure for the Calibration
# of Nutech Model 221 Gas Sampler
RTI/ACS-SOP-367-001

1.0 PRINCIPLE

All Nutech sampling pumps used in the collection of breath samples must be calibrated in the laboratory using a West Test Meter (W.T.M.) within one month prior to any field sampling operation and within one month after completion of sampling. The correction factor determined for each pump is then used to calculate the actual volume of breath drawn through the Tenax cartridge.

2.0 MATERIALS

2.1 Nutech Model 221 Sampling Pumps
2.2 Male Quick-Connects
2.3 Latex tubing (1/4" i.d.)
2.4 Pasteur pipets, 5 cm lengths
2.5 Indicating Drierite
2.6 Tygon tubing (1/4" i.d.)
2.7 Teflon tape
2.8 Thermogreen septa, Supelco #2-0668
2.9 Stainless steel needles, 18 ga. (1 1/2")
2.10 Mercury thermometer, °C
2.11 Barometer
2.12 Table of Vapor Pressure H_2O vs. °C

3.0 PROCEDURE

3.1 Preparation of the Nutech Pump

3.1.1 Open the back of the pump and check all tubing connections.
3.1.2 Secure any loose connections and replace damaged tubing.

3.1.3 Fill the vapor trap with indicating Drierite.

3.1.4 Seal the vapor trap caps with Teflon tape.

3.1.5 Secure the back of the pump.

3.1.6 Flip the AC power to "off" and the DC power switch to "on".

3.1.7 Attach a sampling line to the Nutech inlet using a male Quick Connect fitting.

NOTE: The sampling line is identical to the tubing and fittings used in the collection of field samples. Its configuration is shown in Figure 1.

3.1.8 Flip the AC power switch to the "on" position.

3.1.9 Using the needle valve mounted on the Nutech pump, adjust the flow to a rate which produces a back pressure of approximately 10 (in Hg) as seen on the Nutech pressure gauge.

NOTE: With the sampling line in place, a back pressure of 10 (in. Hg) closely approximate the rate at which actual breath samples are collected. In order to obtain an accurate correction factor, the Nutech must be calibrated close to actual sampling conditions. Significantly faster or slower flow rates often produce erroneous correction factors.

3.1.10 Flip the AC power switch to the "off" position.

3.1.11 Remove the sampling line and attach the Nutech to the W.T.M. as shown in Figure 2 using a male Quick Connect fitting and open ended tubing.

3.2 Calibration with the West Test Meter

3.2.1 Record the ambient temperature in °C on Report Form.

3.2.2 Record the barometric pressure in (mm Hg) on Report Form.

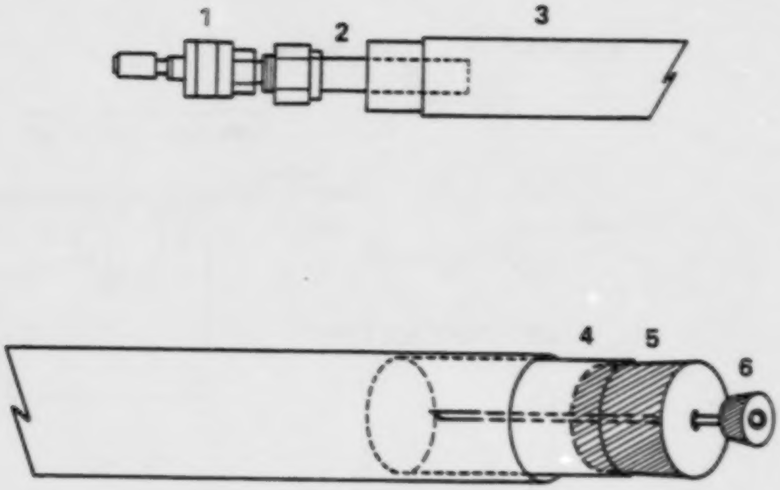
3.2.3 Determine the vapor pressure of water at the ambient temperature from vapor pressure tables.

3.2.4 Record the volume reading from the Nutech dry gas meter.

3.2.5 Record the volume reading from the dial of the W.T.M.

3.2.6 Flip the AC power switch to the "on" position.

3.2.7 Allow the Nutech pump to operate until 1.0-3.0 cu. ft.



1 Quick-Connect fitting
2 ¼" Teflon tubing
3 ¼" i.d. latex tubing
4 ¼" i.d. glass pipet
5 Thermogreen septa
6 18 gal, 1½" stainless steel needle

Figure 1. Sampling Line and Fittings (not to scale)

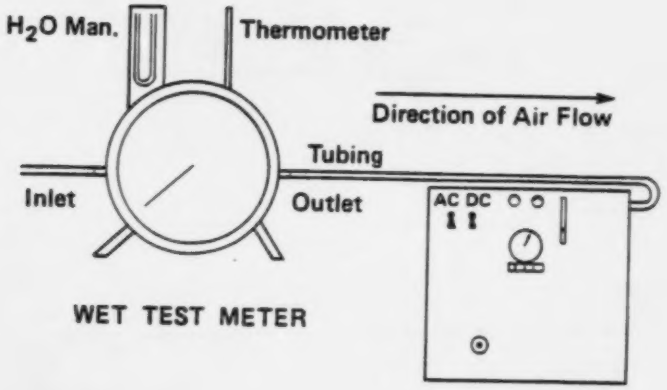

H_2O Man.
Thermometer
Direction of Air Flow
Tubing
Inlet
Outlet
AC DC
WET TEST METER

Figure 2. Nutech Wet Test Meter Configuration

of air has been sampled as determined from the W.T.M. gauge.

3.2.8 While the Nutech pump is in operation, record the difference in water levels, ΔP, observed on the manometer of the W.T.M.

3.2.9 Flip the AC power switch to the "off" position.

3.2.10 Record the final volume readings from the Nutech pump and W.T.M.

3.2.11 Calculate the volume of air sampled as recorded on both the Nutech pump and the W.T.M.

3.3 Calculation of the Correction Factor

3.3.1 Calculate the actual volume of the W.T.M. and the Nutech pump by using equations (1) and (2), respectively.

NOTE: Equations (1) and (2) make adjustments for the pressure drops occurring in the flow meters. Volumes are calculated adjusting for these pressure drops and are adjusted to standard laboratory conditions of temperature and pressure (T_{std} = 298.16 °K, P_{std} = 760.0 mm Hg).

3.2.2 Calculate the correction factor for the Nutech pump using equation (3).

3.3.3 Repeat the calibration procedure twice under the same conditions.

3.3.4 Calculate the mean correction factor from the three individual measurements.

3.3.5 Record the mean correction factor, to two significant figures, the date, and your initials on the front label of the Nutech pump and in a laboratory log notebook.

NOTE: The mean correction factor should fall in the range 0.9-1.1. If the calculated value falls outside of this range, do not use the pump for the breath collection unless approved by Task Leader.

3.3.6 Send results of pump calibration to Task Leader and QA Officer.

Calibration of Nutech Model 221 Gas Sampler

REPORT FORM

Date: ____________ Pump No.:______________ Analyst: ____________________________

Temperature (°C): __________ Pressure (mm Hg): _____________ Vapor Pressure (H_2O):______

Manometer Reading (ΔP) on Wet Test Meter (inches H_2O): ______________

Volume Reading from Wet Test Meter (W.T.M.):

Final = _____ W.T.M. (b) = _____

Initial = ________ W.T.M. (M) = _____

Net (Q_{ind}) = __________

Correction of W.T.M. Volume:

$$Q_{act} = \left[\frac{\left[Q_{ind} \left[\frac{P - VP - (\Delta P \text{ X } D)}{760} \right] \frac{298.15}{273.15 + T} \right]}{M} \right] - b$$

Q_{act} = Actual volume measured by Wet Test Meter.

Q_{ind} = Volume indicated by Wet Test Meter

P = Barometric pressure (mm Hg)

VP = Vapor pressure of water at ambient temperature (mm Hg)

ΔP = Water manometer reading on W.T.M. (inches H_2O)

D = Conversion factor: 25.4 mm Hg/13.5 s.g. Hg.

T = Temperature, °C

M,b = W.T.M. constants

Q_{act} = __________________

Correction of W.T.M. Volume:

$$Q'_{act} = Q'_{ind} \left[\frac{P}{760} \text{ X } \frac{298.15°K}{273.15°K + T} \right]$$

Q'_{act} = Actual volume measured by Nutech meter.

Q'_{ind} = Volume indicated by Nutech meter.

Q'_{act} = __________________

Correction Factor Nutech Pump No. ______ = Q_{act}/Q'_{act} = ____________

Reference: SOP for Calibration of Nutech Model 221 Gas Sampler

Analyst: ____________________________

Equation (1)

$$Q_{act} = \left[\frac{\left[Q_{ind} \left[\frac{\text{B.P. (mm Hg)} - \text{V.P.}_{H_2O\ amb} - (\Delta P_{H_2O\ amb} \times D)}{760\ \text{mm Hg}} \right] \times \frac{298.15}{273.15 + {}^\circ C} \right]}{M} \right] - b$$

Q_{act} = Actual volume measured by Wet Test Meter

Q_{ind} = Volume indicated by Wet Test Meter

B.P. = Barometric pressure, mm Hg

V.P. = Vapor pressure of H_2O at ambient temperature, mm Hg

ΔP = Difference in H_2O manometer for W.T.M., H_2O (in)

D = Conversion factor from (in) H_2O to (mm) Hg: (25.4 mmHg/13.546 s.g. Hg)

M = Intercept of W.T.M. calibration curve: listed in specific W.T.M.

b = Slope of W.T.M. calibration curve: listed on specific W.T.M.

Equation (2)

$$Q'_{act} = Q'_{ind} \left[\frac{\text{B.P. mm Hg}}{760\ \text{mmHg}} \times \frac{298.15^\circ K}{273.15^\circ K + {}^\circ C} \right]$$

Q'_{act} = Actual volume measured by Nutech dry gas meter.

Q_{ind} = Volume indicated by Nutech dry gas meter

Equation (3)

$$\frac{Q_{act}}{Q'_{act}} = \text{Correction Factor}$$

RTI/ACS-SOP-367-002
January 1984
Revision 0

Standard Operating Procedure for the Calibration of Nutech Model 221 Gas Sampler Using a Dry Gas Meter

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Standard Operating Procedure for the Calibration of Nutech Model 221 Gas Sampler Using a Dry Gas Meter

RTI/ACS-SOP-367-002

1.0 PRINCIPLE

All Nutech sampling pumps used in the collection of breath samples must be calibrated in the laboratory using a Dry Gas Meter (D.G.M.) within one month prior to any field sampling operation and within one month after completion of sampling. No corrections due to back pressure changes, barometric pressure, or actual temperature are required since the meter on the Nutech measures actual volume passing through the pump. The correction factor determined for each pump is then used to calculate the actual volume of breath drawn through the Tenax cartridge.

This procedure is an alternative method for calibrating the Nutech sampling pumps. The use of a Wet Test Meter is described in RTI/ACS-SOP-367-001.

2.0 MATERIALS

2.1 Nutech Model 221 Sampling Pumps
2.2 Male Quick-Connects
2.3 Latex tubing (1/4" i.d.)
2.4 Pasteur pipets, 5 cm lengths
2.5 Indicating Drierite
2.6 Tygon tubing (1/4" i.d.)
2.7 Teflon tape
2.8 Thermogreen septa, Supelco #2-0668
2.9 Stainless steel needles, 21 ga. (1 1/2")

3.0 PROCEDURE

3.1 Preparation of the Nutech Pump

3.1.1 Open the back of the pump and check all tubing connections.

3.1.2 Secure any loose connections and replace damaged tubing.

3.1.3 Fill the vapor trap with indicating Drierite.

3.1.4 Seal the vapor trap caps with Teflon tape.

3.1.5 Secure the back of the pump.

3.1.6 Flip the AC power to "off" and the DC power switch to "on".

3.1.7 Attach a sampling line to the Nutech inlet using a male Quick Connect fitting.

NOTE: The sampling line is identical to the tubing and fittings used in the collection of field samples. Its configuration is shown in Figure 1.

3.1.8 Flip the AC power switch to the "on" position.

3.1.9 Using the needle valve mounted on the Nutech pump, adjust the flow to a rate which produces a back pressure of approximately 5 inches Hg as seen on the Nutech pressure gauge.

NOTE: With the sampling line in place, a back pressure of 5 (in. Hg) closely approximate the rate at which actual breath samples are collected. In order to obtain an accurate correction factor, the Nutech must be calibrated close to actual sampling conditions. Significantly faster or slower flow rates often produce erroneous correction factors.

3.1.10 Flip the AC power switch to the "off" position.

3.1.11 Remove the sampling line and attach the Nutech to the D.G.M. as shown in Figure 2 using a male Quick Connect fitting and open ended tubing.

3.2 Calibration with the West Test Meter

3.2.1 Record the ambient temperature in °C on Report Form.

3.2.2 Record the volume reading from the Nutech dry gas meter.

3.2.3 Record the volume reading from the dial of the D.G.M.

3.2.4 Flip the AC power switch to the "on" position.

3.2.5 Allow the Nutech pump to operate until 5.0-10.0 Liters of air has been sampled as determined from the D.G.M. gauge.

3.2.6 While the Nutech pump is in operation, record the back pressure, ΔP, observed on the manometer of the Nutech.

3.2.7 Flip the AC power switch to the "off" position.

3.2.8 Record the final volume readings from the Nutech pump and D.G.M.

3.2.9 Calculate the volume of air sampled as recorded on both the Nutech pump and the D.G.M.

3.3 Calculation of the Correction Factor

3.3.1 Calculate the actual volume of the D.T.M. and the Nutech pump by using equations (1) and (2), respectively.

3.2.2 Calculate the correction factor for the Nutech pump using equation (3).

3.3.3 Repeat the calibration procedure twice under the same conditions.

3.3.4 Calculate the mean correction factor from the three individual measurements.

3.3.5 Record the mean correction factor, to two significant figures, the date, and your initials on the front label of the Nutech pump and in a laboratory log notebook.

NOTE: The mean correction factor should fall in the range 0.9-1.1. If the calculated value falls outside of this range, do not use the pump for the breath collection unless approved by Task Leader.

3.3.6 Send results of pump calibration to Task Leader and QA Officer.

Equation (1)

$$Q_{act} = Q_{ind}$$

Q_{act} = Actual volume measured by Dry Gas Meter

Q_{ind} = Volume indicated by Dry Gas Meter

Equation (2)

$$Q'_{act} = Q'_{ind} \cdot L$$

Q'_{act} = Actual volume measured by Nutech dry gas meter.

Q_{ind} = Volume indicated by Nutech dry gas meter

L = Conversion factor from cu. ft. to Liters: (28.3 Liters/cu. ft.)

Equation (3)

$$\frac{Q_{act}}{Q'_{act}} = \text{Correction Factor}$$

REPORT FORM

Calibration of Nutech Model 221 Gas Sampler

Date: ____________ Pump No.: ____________ Analyst: ____________

Temperature (°C): ________________

Manometer Reading (ΔP) on Nutech (in) Hg: ______________

Triplicate Measurements

Volume Reading from Dry Gas Meter (D.G.M.):

Final = ____________, ____________, ____________

Initial = ____________, ____________, ____________

Net (Q_{act}) = ____________, ____________, ____________

Q_{act} = Actual volume measured by Dry Gas Meter (Liters)

Correction of Nutech Volume:

$$Q'_{act} = Q'_{ind} \quad L$$

Q'_{act} = Actual volume measured by Nutech meter (Liters)

L = Conversion factor: 28.3 Liters/cu. ft.

Q'_{ind} = Volume indicated by Nutech meter (cu. ft.)

Q'_{act} = ____________, ____________, ____________

Correction Factor (Q_{act}/Q'_{act}) = ____________, ____________, ____________

Mean Correction Factor: ________________

Reference: SOP for Calibration of Nutech Model 221 Gas Sampler.

RTI/ACS-SOP-410-001
December 1983
Revision 0

Standard Operating Procedure for Using Sampling Protocol/ Chain-of-Custody Sheets in the Field

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Standard Operating Procedure for Using Sampling Protocol/ Chain-of-Custody Sheets in the Field

RTI/ACS-SOP-410-001

1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is to describe the proper way in which Field Sampling Protocol/Chain-of-Custody (SP/COC) Sheets must be filled out in the field. An SP/COC sheet must be generated for every sample collected in the field and accompany every quality control (QC) sample carried to/from the field. The procedures documented herein pertain specifically to Tenax (personal air, fixed-site air, and breath) samples and water samples. The SP side may be modified to accommodate any type of discrete sample collected. The COC record requires no modification in its use with different types of samples.

2.0 SUMMARY OF THE METHOD

The significance of the SP/COC sheet is two-fold: (1) the SP side (front) documents information about the specific conditions associated with sample collection; and (2) the COC side (back) provides a record of each operation performed on the sample, the date, and person responsible for that activity, including receipt and transmittal of the sample.

When a sample is collected, the SP sheet is filled in. The sheet contains a unique sample code identifying the project, year, season, site, respondent number, check digit, matrix, sample type, and sampling period. Also included are sampling times, volume(s) collected, meteorology and other pertinent information (see Figure 1). Where appropriate, a map is made to identify the precise location of sampling.

Each sample is accompanied by a COC record which is maintained from the time of collection (or QC sample preparation) through reporting of analytical results (see Figure 2). This record is important for identifying sample contamination, a questionable standard, or some other problem.

3.0 PROCEDURE

3.1 Filling Out a Field Sampling Protocol Sheet-TEAM Study

3.1.1 Date

3.1.1.1 For a field sample, enter the date sample collection was started.

3.1.1.2 For a QC sample, enter the date of preparation.

3.1.2 Code for Samples

3.1.2.1 Use "TEAM" as the project identifier.

3.1.2.2 Use a six-digit code to identify the year, season, respondent number and check digit, followed by the codes for matrix, sample type and visit number.

NOTE: (1) Self-adhesive labels bearing the project identifier and the six-digit code are prepared by the Computer Applications Center (CAC) prior to the sampling trip. Only the matrix, sample type and visit number must be filled in by the person collecting the sample. The respondent numbers are as follows:

001-399	Elizabeth, NJ
400-699	Bayonne, NJ
700-750	Greensboro, NC
800-850	Devil's Lake, ND

(2) The check digit following the respondent number is determined from a series of mathematical calculations performed on the five other digits of the code. The check-digit is used only by CAC to verify the correctness of the sample code.

3.1.2.3 Use the following abbreviations for matrix sampls.

AV - personal air volatiles
XV - fixed site volatiles
BR - breath
WV - water volatiles

FIELD SAMPLING PROTOCOL SHEET - TEAM STUDY

DATE: ______________________________

SAMPLE CODE: ____

AIR SAMPLING

Pump Model: __________________ Serial Number: ____________________

Time (Initial): ______________ Flow Rates (Initial): _____________

(Final): ______________ (Final): _____________

Total Time: __________________ Avg. Flow Rate: ___________________

Total Volume Sampled: __________________

Temperature (°F): ______________________

BREATH SAMPLING

Meter Readings Final: __________ft^3 Time of Day: __________________

Initial: __________ft^3 Temperature: __________________°F

Total: __________ft^3 Duration of Exhalation __min__sec

Correction Factor: X ____________ Pump No.: ______________________

Volume/Cartridge: __________________L (1 ft^3 = 28.3 L)

DRINKING WATER, BLOOD, URINE, MILK SAMPLING

Time of Day: __________________ Source (water only) _____________
(e.g. ground, city, etc.)

Amount Collected: _____________

FOOD SAMPLING

Time Period During Which Food was Consumed: ______________________

Time of Collection: ____________

Container	Container Type	Description of Food Type
(1)	______________	______________________
(2)	______________	______________________
(3)	______________	______________________

GENERAL REMARKS

Figure 1. The sampling protocol sheet for each sample.

CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute, Analytical Sciences Division
Chemistry and Life Sciences Group, Research Triangle Park, NC 27709

SAMPLE CODE	TENAX BATCH:	VOLUME COLLECTED:
SAMPLE TYPE:	TEMPERATURE (°F)	VOLUME ANALYZED:

RELIN-QUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED

Figure 2. The chain of custody record for each sample.

3.1.2.4 Use the following abbreviations for sample type collected:

F - field
D - duplicate
Q - quality assurance

3.1.2.5 Use the digits 1, 2, or 3 to indicate the period of collection if the sample is collected at more than one period.

3.1.2.6 See Examples 1, 2, and 3 (Figure 3) for determining appropriate field sample codes for water, air, and breath samples, respectively.

3.1.3 Code for Blanks and Controls

3.1.3.1 Use "TEAM" as the project identifier.

3.1.3.2 Use a four-digit code to identify year, season and site, followed by the codes for matrix, sample type and QC number.

NOTE: Self-adhesive labels bearing the project identifier and the four-digit code are prepared by CAC prior to the sampling trip. The matrix, sample type and QC number are added by the staff member responsible for the preparation of QC samples. The site numbers are as follows:

6 - Devil's Lake, ND
7 - Greensboro, NC
8 - Elizabeth, NJ
9 - Bayonne, NJ

3.1.3.3 Use the following abbreviations for matrix sampled.

AV - personal air volatiles
XV - fixed-site volatiles
BR - breath
WV - water volatiles

3.1.3.4 Use the following abbreviations for sample type exposed:

FB - field blank
SB - spirometer blank
FC - field control

Example 1: A duplicate water sample collected during the third visit to a Bayonne respondent during the first season of the second year.

TEAM	2	1	4 1 0	-	6	WV	-	D	3
Project Identifier	Year	Season	Respondent Number		Check Digit	Matrix		Sample Type	Visit Number

Example 2: A personal air field sample collected during the second visit to an Elizabeth respondent during the second season of the first year.

TEAM	1	2	0 7 1	-	7	AV	-	F	2
	Year	Season	Respondent Number		Check Digit	Matrix		Sample Type	Visit Number

Example 3: A QA breath sample collected from a Greensboro respondent during the first season of the first year.

TEAM	1	1	7 0 3	-	6	BR	-	Q*
	Year	Season	Respondent Number		Check Digit	Matrix		Sample Type

*Note that visit number is not required because breath samples are collected only during the third visit.

3.1.3.5 See Examples 4, 5, and 6 (Figure 4) for determining appropriate blank and control codes for water, air and breath samples, respectively.

3.1.4 Air Sampling (Personal and Fixed Site)

3.1.4.1 Enter the model number of the pump used.

3.1.4.2 Enter the serial number of the pump used.

3.1.4.3 Enter the time (military) sampling is initiated.

3.1.4.4 Enter the initial flow rates (mL/min) with three significant figures.

NOTE: For example, the value 28.6 mL/min has three significant figures; the first two are firm and the third figure (6) an estimate (between 5 and 7).

3.1.4.5 Enter the temperature measured at the site when sampling is begun.

3.1.4.6 Enter the time that sampling is terminated.

3.1.4.7 Enter the final flow rate with three significant figures.

3.1.4.8 Enter the temperature measured at the site when sampling is terminated.

3.1.4.9 Compute and enter the total time elapsed (in minutes) during sampling. See Figures 5 and 6.

Example (from Figure 5)

Initial time = 1952
Final time = 0857 (following day)

First, calculate the time sampled during day 1.

$$\begin{array}{rcrl} 2400 \text{ hr} & = & 23 \text{ hr } 60 \text{ min} & \\ & & \underline{-19 \text{ hr } 52 \text{ min}} & \\ & & 4 \text{ hr } \ \ 8 \text{ min} & = \ 248 \text{ min} \end{array}$$

Next, calculate the time sampled during day 2.

0857 hr = 8 hr 57 min = 537 min

Last, add these times to get the total elapsed time.

248 min + 537 min = 785 min

3.1.4.10 Compute and enter the average flow rate with three significant figures (see Figure 5).

$$\begin{array}{rl} 29.1 \text{ mL/min} & \\ \underline{+30.6 \text{ mL/min}} & \\ 59.7 \text{ mL/min}/2 & = 29.8 \text{ mL/min} \end{array}$$

NOTE: The rounding off rule is as follows:
If the number to the right of the last significant figure is:

Example 4: A water field blank for Bayonne, second year, first season.

2	1	9	-	6	WV	-	FB	10
Year	Season	City		Check Digit	Matrix		Sample Type	QC Number

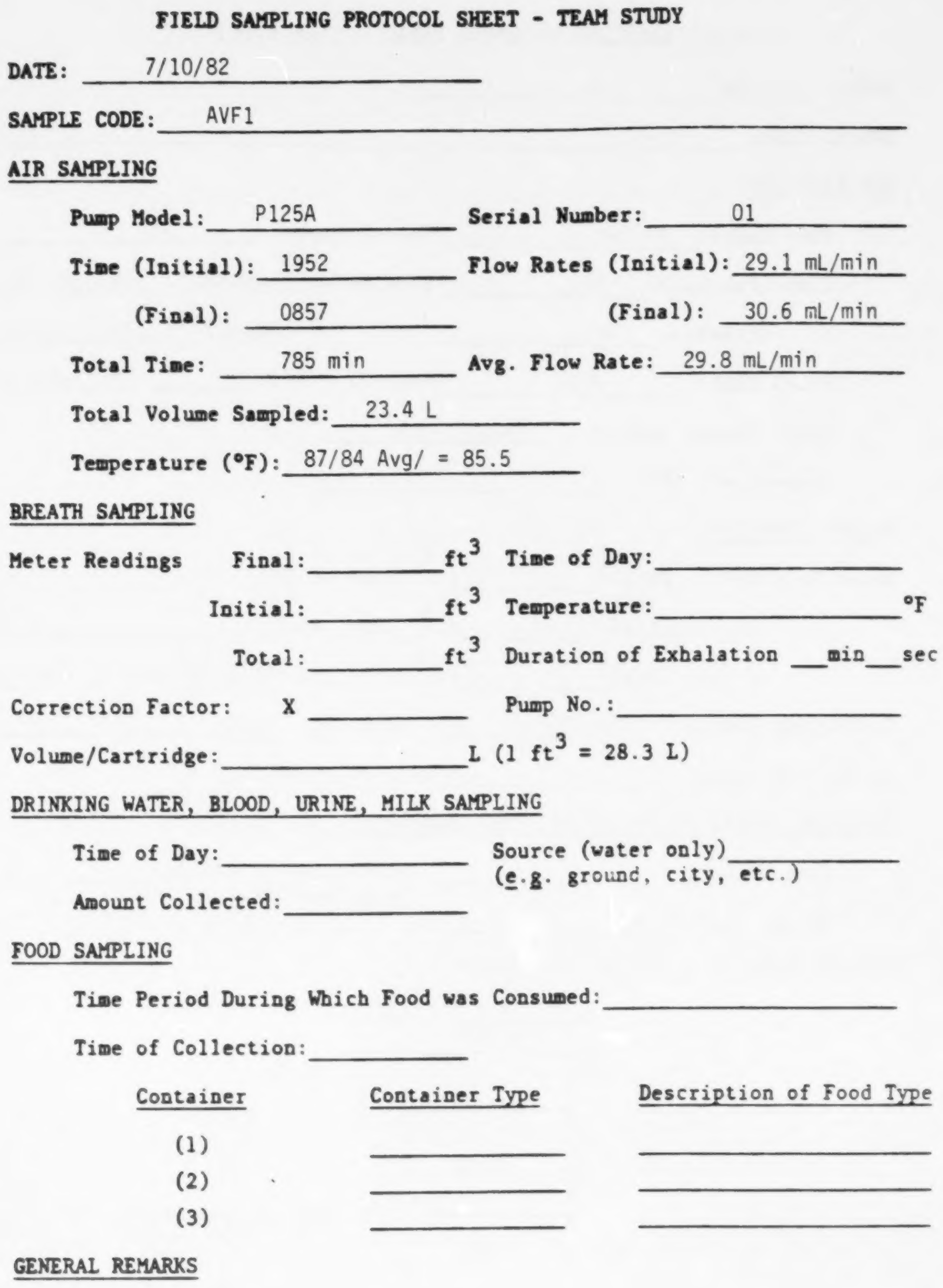
Example 5: A personal air field control for Elizabeth, second year, first season.

2	1	8	8	AV	-	FC	11
Year	Season	City	Check Digit	Matrix		Sample Type	QC Number

Example 6: A breath spirometer blank for Devil's Lake, second year, second season.

2	2	6	-	1	BR	-	SB	4
Year	Season	City		Check Digit	Matrix		Sample Type	QC Number

Figure 4. Examples of TEAM QC Sample Codes



FIELD SAMPLING PROTOCOL SHEET - TEAM STUDY

DATE: 7/10/82

SAMPLE CODE: AVF1

AIR SAMPLING

Pump Model: P125A Serial Number: 01

Time (Initial): 1952 Flow Rates (Initial): 29.1 mL/min

(Final): 0857 (Final): 30.6 mL/min

Total Time: 785 min Avg. Flow Rate: 29.8 mL/min

Total Volume Sampled: 23.4 L

Temperature (°F): 87/84 Avg/ = 85.5

BREATH SAMPLING

Meter Readings Final: ______ ft^3 Time of Day: ______

Initial: ______ ft^3 Temperature: ______ °F

Total: ______ ft^3 Duration of Exhalation __min__sec

Correction Factor: X ______ Pump No.: ______

Volume/Cartridge: ______ L (1 ft^3 = 28.3 L)

DRINKING WATER, BLOOD, URINE, MILK SAMPLING

Time of Day: ______ Source (water only) ______
(e.g. ground, city, etc.)

Amount Collected: ______

FOOD SAMPLING

Time Period During Which Food was Consumed: ______

Time of Collection: ______

Container	Container Type	Description of Food Type
(1)		
(2)		
(3)		

GENERAL REMARKS

Figure 5. Completed SP sheet for a personal air sample.

FIELD SAMPLING PROTOCOL SHEET - TEAM STUDY

DATE: 7/12/82

SAMPLE CODE: XV-F2

AIR SAMPLING

Pump Model: P125A **Serial Number:** 36

Time (Initial): 801 **Flow Rates (Initial):** 26.3 mL/min

(Final): 1604 **(Final):** 26.0 mL/min

Total Time: 483 **Avg. Flow Rate:** 26.2 mL/min

Total Volume Sampled: 12.6 L

Temperature (°F): ____

BREATH SAMPLING

Meter Readings Final: ____ ft^3 **Time of Day:** ____

Initial: ____ ft^3 **Temperature:** ____ °F

Total: ____ ft^3 **Duration of Exhalation** __ min __ sec

Correction Factor: X ____ Pump No.: ____

Volume/Cartridge: ____ L (1 ft^3 = 28.3 L)

DRINKING WATER, BLOOD, URINE, MILK SAMPLING

Time of Day: ____ Source (water only) ____
(e.g. ground, city, etc.)

Amount Collected: ____

GENERAL REMARKS Site Description

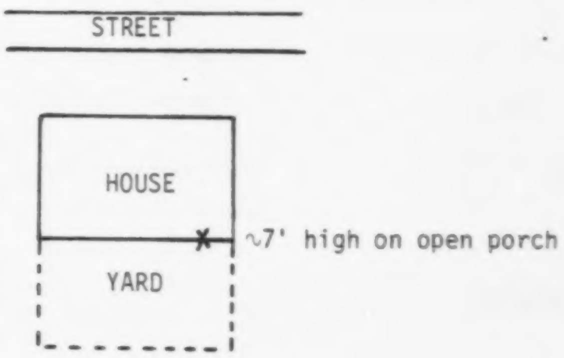


Figure 6. Completed SP sheet for a fixed-site sample.

< 5, drop the number
> 5, add 1 to the last significant figure
= 5, drop it if the last significant figure is an even number or add 1 to the last significant figure if it is an odd number.

NOTE: Other examples:

Initial/Final Rates	Average	Round to
28.3; 28.6	28.45	28.4
25.4; 25.7	25.55	25.6

3.1.4.11 Compute and enter the total volume sampled (L) with three significant figures (see Figure 5).

785 min x 29.8 mL/min = 23,393 mL = 23.4 L

NOTE: Other examples:

Sampling Time (min)	Average Rate (mL/min)	Sample Volume (L) Calcd	 Enter
700	28.4	19.88	19.9
800	25.6	20.48	20 5

3.1.4.12 Compute and enter the average temperature (i.e., initial plus final temperature/2).

3.1.5 Breath Sampling

3.1.5.1 Enter the initial meter reading on the Nutech pump to the nearest 0.001 ft.^{3}.

3.1.5.2 Enter the pump number and correction factor for that pump.

3.1.5.3 Enter the time of sample collection.

3.1.5.4 Enter the temperature in the van when the breath sample collection occurs.

3.1.5.5 Start the timer when the respondent begins to provide sample.

3.1.5.6 Stop the timer when the respondent has completed providing sample and note the duration of exhalation on the SP sheet.

3.1.5.7 Enter the final meter reading (to the nearest 0.001 ft.^{3}) after the sample has been pumped onto the Tenax cartridge.

3.1.5.8 Calculate and enter the total volume of air sampled to the nearest 0.001 ft.^{3}.

3.1.5.9 Multiply the total volume by the correction factor for that pump.

3.1.5.10 Convert ft.^{3} to liters (L) by multiplying by 28.3 L/ft.^{3} and enter this value with three significant figures for Volume/Cartridge (see Figure 7).

NOTE: The rounding-off rule defined in Section 3.1.4.10 will be used.

3.1.6 Water Sampling

3.1.6.1 Circle "Drinking Water" on the SP sheet (see Figure 8).

3.1.6.2 Record the time of day when the sample is collected.

3.1.6.3 Record the amount collected.

3.1.6.4 Record the source of water supply and location where it was obtained (e.g., kitchen tap, drinking fountain, bottled water).

3.1.6.5 See Figure 8 for an example of a completed SP sheet for water.

3.1.7 General Remarks

3.1.7.1 In this section of the SP sheet, include any of the following information: (1) details of the sampling that are nonroutine (e.g., a respondent that had to be sampled at his workplace); (2) a map depicting the location of a fixed-site sampler; (3) mention of any instrumental problems encountered during collection (e.g., pump failure); and (4) any action by the respondent or sampling staff that might invalidate the sample (e.g., handling Tenax cartridge with fingers).

3.2 Filling Out a Chain-of-Custody Record (TEAM Study)

3.2.1 Sample Code

3.2.1.1 Affix a preprinted label in this box.

3.2.1.2 Add the appropriate matrix, sample type and visit codes, ensuring that the complete code on the SP side matches the code on the COC side.

3.2.2 Tenax Batch

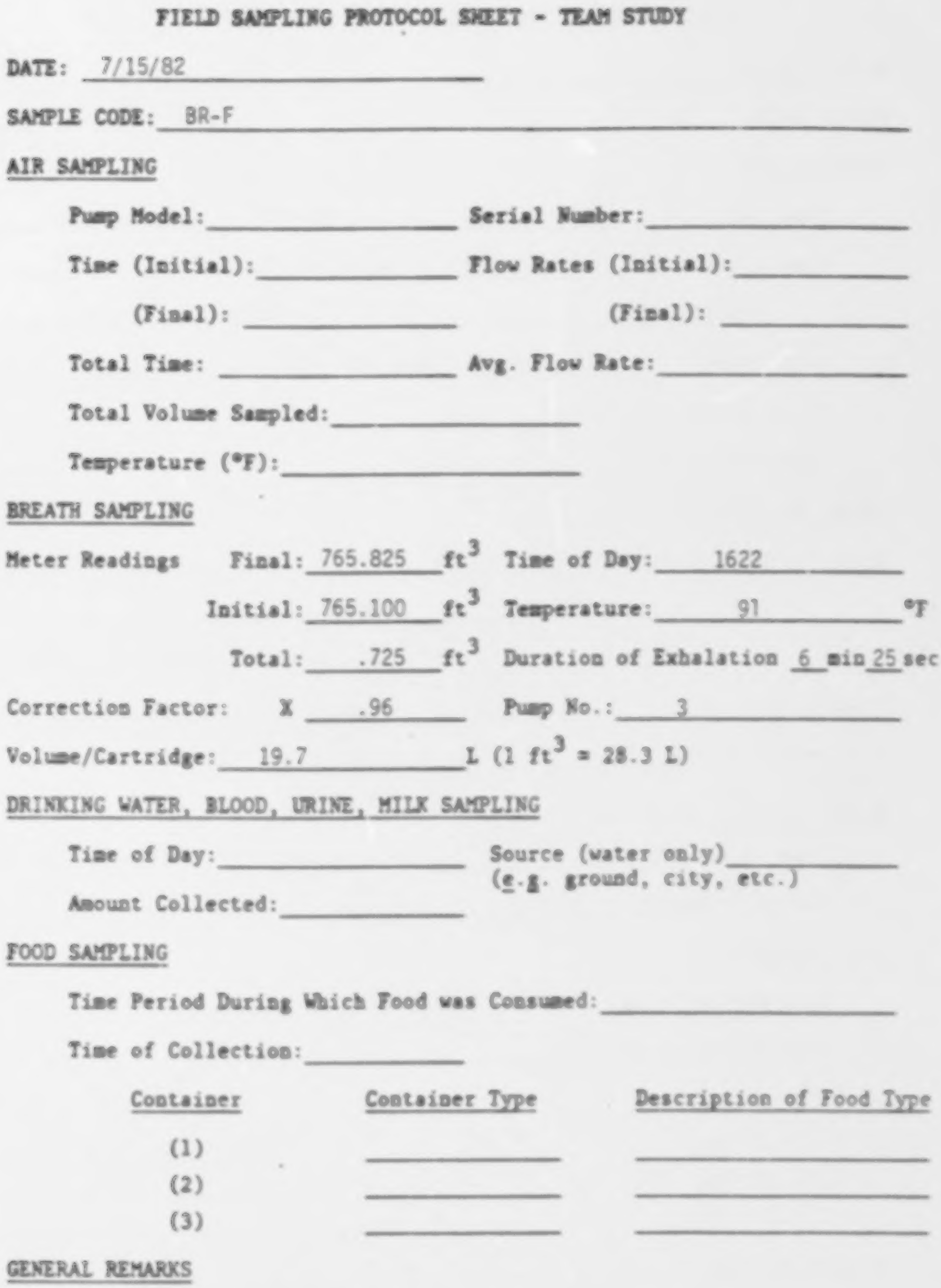
3.2.1.1 Fill in this box for Tenax samples only.

3.2.1.2 Record the batch number located on the screw cap of the Kimax tube containing the Tenax cartridge used.

3.2.3 Sample Type

3.2.3.1 Enter "TENAX" or "water".

NOTE: The abbreviations T_x or H_2O may be used for Tenax and water, respectively.



FIELD SAMPLING PROTOCOL SHEET - TEAM STUDY

DATE: 7/15/82

SAMPLE CODE: BR-F

AIR SAMPLING

Pump Model: ______ Serial Number: ______

Time (Initial): ______ Flow Rates (Initial): ______

(Final): ______ (Final): ______

Total Time: ______ Avg. Flow Rate: ______

Total Volume Sampled: ______

Temperature (°F): ______

BREATH SAMPLING

Meter Readings Final: 765.825 ft^3 Time of Day: 1622

Initial: 765.100 ft^3 Temperature: 91 °F

Total: .725 ft^3 Duration of Exhalation 6 min 25 sec

Correction Factor: X .96 Pump No.: 3

Volume/Cartridge: 19.7 L (1 ft^3 = 28.3 L)

DRINKING WATER, BLOOD, URINE, MILK SAMPLING

Time of Day: ______ Source (water only) ______ (e.g. ground, city, etc.)

Amount Collected: ______

FOOD SAMPLING

Time Period During Which Food was Consumed: ______

Time of Collection: ______

Container	Container Type	Description of Food Type
(1)		
(2)		
(3)		

GENERAL REMARKS

Figure 7. Completed SP sheet for a breath sample.

FIELD SAMPLING PROTOCOL SHEET - TEAM STUDY

DATE: 7/8/82

SAMPLE CODE: WV-F2

AIR SAMPLING

Pump Model: ________ Serial Number: ________

Time (Initial): ________ Flow Rates (Initial): ________

(Final): ________ (Final): ________

Total Time: ________ Avg. Flow Rate: ________

Total Volume Sampled: ________

Temperature (°F): ________

BREATH SAMPLING

Meter Readings Final: ________ ft^3 Time of Day: ________

Initial: ________ ft^3 Temperature: ________ °F

Total: ________ ft^3 Duration of Exhalation __ min __ sec

Correction Factor: X ________ Pump No.: ________

Volume/Cartridge: ________ L (1 ft^3 = 28.3 L)

[✓ DRINKING WATER,] BLOOD, URINE, MILK SAMPLING

Time of Day: 722 Source (water only) Kitchen tap
(e.g. ground, etc.)

Amount Collected: 60 mL

FOOD SAMPLING

Time Period During Which Food was Consumed: ________

Time of Collection: ________

Container	Container Type	Description of Food Type
(1)		
(2)		
(3)		

GENERAL REMARKS

Figure 8. Completed SP sheet for a water sample.

3.2.4 Temperature

3.2.4.1 For a breath sample, enter the temperature at the time of collection. Ensure that this temperature matches that recorded on the SP side.

3.2.4.2 For a personal air sample, enter the average temperature (°F) during collection as calculated on the SP side.

3.2.4.3 Do not make any temperature entries for fixed-site or water samples.

NOTE: Temperatures used for fixed-site samples are calculated from data provided by the Weather Service serving the particular city sampled.

3.2.5 Volume Collected

3.2.5.1 For Tenax samples, enter the total volume collected (in L) which was calculated on the SP side.

3.2.5.2 For water samples, enter the volume collected (in mL) as indicated on the SP side.

3.2.6 Custody Record During Air Sample Collection (Personal and Fixed Site)

3.2.6.1 At the start of sample collection, enter your initials in the "Rec'd By" column, the time sample collection began, the date, and the operation performed (i.e., Tenax exposed). Ensure that the time and date entered match the entries on the SP side.

NOTE: The abbreviation TE may be used for Tenax exposed.

3.2.6.2 Enter your initials in the "Relinquished By" column.

3.2.6.3 At the end of sample collection, enter your initials in the "Rec'd By" column, the time that sample collection was terminated, the date and operation performed (i.e., Tenax collected).

NOTE: The abbreviation TC may be used for Tenax collected.

3.2.6.4 Record your initials in the "Relinquished By" column.

3.2.7 Custody Record for Breath and Water Sample Collection

3.2.7.1 For sample collection, enter your initials in the "Rec'd By" column, the time the sample collection was terminated, the date and the operation performed (i.e., sample collected). Ensure that the time and date entered match the entries on the SP side.

NOTE: The abbreviation SC may be used for sample collected.

3.2.7.2 Enter your initials in the "Relinquished By" column.

3.2.8 Custody Record After Sample Collection

For any operation performed on the sample (e.g., QA check, shipment and receipt of samples), complete an entry on the COC record. Include your initials, date and operation performed. See Figures 9, 10, 11, and 12 for COC records of personal, fixed-site, breath, and water samples, respectively.

CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute, Analytical Sciences Division
Chemistry and Life Sciences Group, Research Triangle Park, NC 27709

SAMPLE CODE: TEAM 12135: AV-F2	**TENAX BATCH:** 35	**VOLUME COLLECTED:** 20.9L
SAMPLE TYPE: TX	**TEMPERATURE: (°F)** 68	**VOLUME ANALYZED:**

RELIN-QUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	JH	6.30	11/3/81	TE
JH	JH	1800	11/3 81	TC
JH	[illegible]		11/3/81	QA
[illegible]	BWI		NOV 05 1981	SENT TO RTI VIA FEDERAL EXPRESS
[illegible]	PAB		NOV 06 1981	RECEIVED AT RTI
PAB	NC		FEB 11 1982	PFB 128 PFT 124 TO MS 3300
NC				

Figure 9. COC record for a personal air sample.

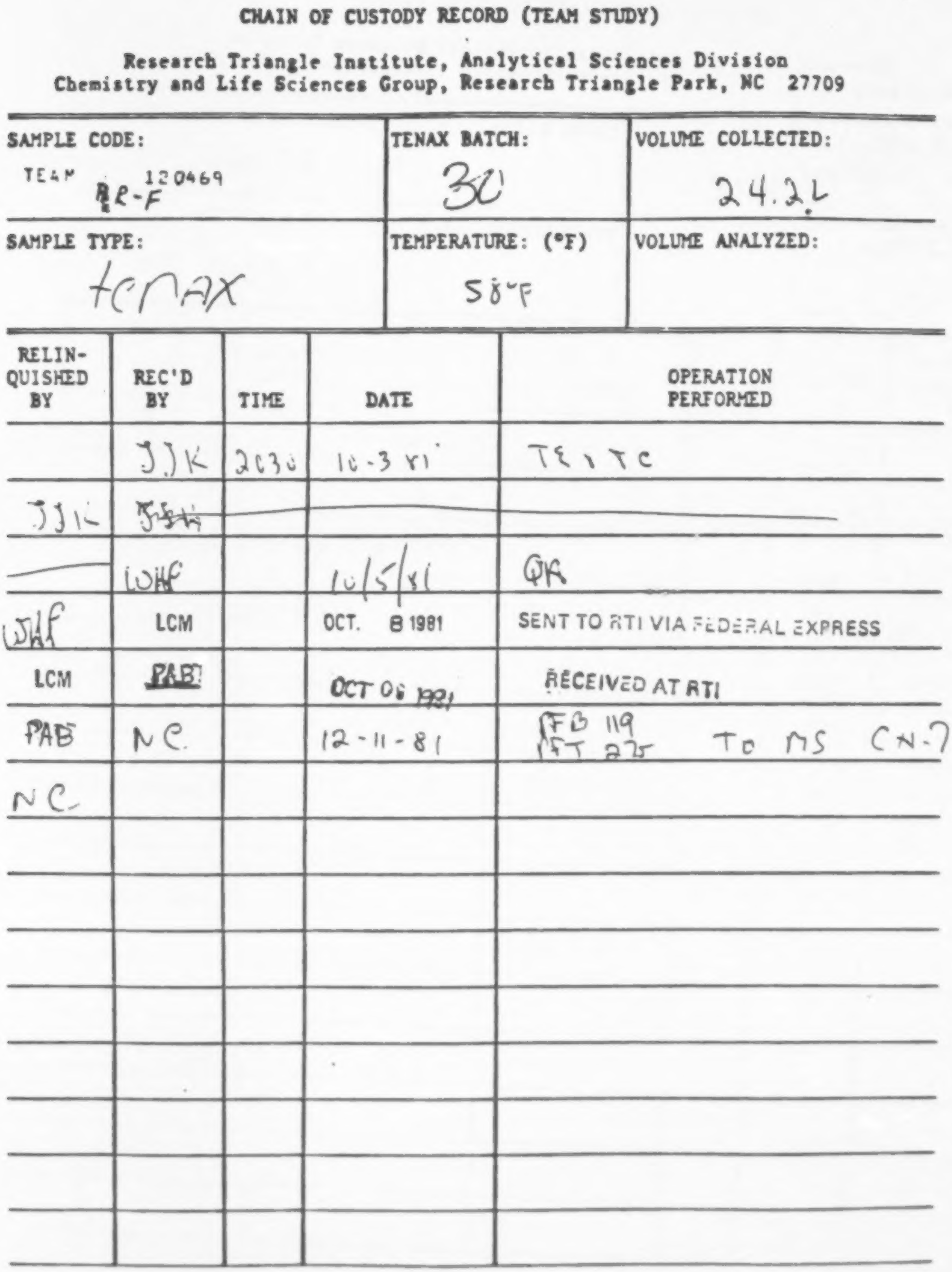
CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute, Analytical Sciences Division
Chemistry and Life Sciences Group, Research Triangle Park, NC 27709

SAMPLE CODE: TEAM 120717 XV-F1	**TENAX BATCH:** 32	**VOLUME COLLECTED:** 23.8L
SAMPLE TYPE: Fixed Site/Tenax	**TEMPERATURE: (°F)**	**VOLUME ANALYZED:**

RELIN-QUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	JL	1911	10/12/81	ET
JL	JL	739	10/13/81	SC
JL	MDW	1320	10/14/81	QA
MDW	[illegible]		OCT 18 1981	SENT TO RTI VIA FEDERAL EXPRESS
[illegible]	PAB		OCT 20 1981	RECEIVED AT RTI
PAB	NC		JAN 5 1982	PFB 125 PFT 271 To MS CH-7
NC				

Figure 10. COC record for a fixed-site air sample.



CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute, Analytical Sciences Division
Chemistry and Life Sciences Group, Research Triangle Park, NC 27709

SAMPLE CODE: TEAM 120469 BR-F	TENAX BATCH: 30	VOLUME COLLECTED: 24.2L
SAMPLE TYPE: tenax	TEMPERATURE: (°F) 58°F	VOLUME ANALYZED:

RELIN-QUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	JJK	2030	10-3-81	TE 1 TC
JJK	~~JJK~~			
	WHF		10/5/81	QR
WHF	LCM		OCT. 8 1981	SENT TO RTI VIA FEDERAL EXPRESS
LCM	PAB		OCT 06 1981	RECEIVED AT RTI
PAB	NC.		12-11-81	FB 119 FT 275 TO MS CN-7
NC				

Figure 11. COC record for a breath sample.

CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute, Analytical Sciences Division
Chemistry and Life Sciences Group, Research Triangle Park, NC 27709

SAMPLE CODE: TEAM 120014 WVF2	**TENAX BATCH:**	**VOLUME COLLECTED:** 60 ml
SAMPLE TYPE: Water	**TEMPERATURE: (°F)**	**VOLUME ANALYZED:** 25 ml

RELIN-QUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	DAJ	0705	9/19/81	SC
DAJ	MMF	1205	9-19-81	QA
MMF	RWH		SEP 30 1981	SENT TO RTI VIA FEDERAL EXPRESS
RWH	PAB		OCT 01 1981	RECEIVED AT RTI
PAB			10/22/81	P+T Analysis
	FM		2/3/82	Transcribed
FM	PAB		FEB 05 1982	QA
PAB				

Figure 12. COC record for a water sample.

RTI/ACS-SOP-431-001 (Air)
432-001 (Water)
437-001 (Breath)
April 1984
Revision 1

Standard Operating Procedure for the Storage of Samples at the Field Sampling Site

Author:

Patricia A. Blau
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Approved:

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Laboratory Manager, RTI/ACS — 5/3/84 Date

RW Handy
QA Officer, RTI/ACS — 4/13/84 Date

E Pellizzari
Vice-President, RTI/ACS — 5/3/84 Date

Standard Operating Procedure for the Storage of Samples at the Field Sampling Site

RTI/ACS-SOP-431-001 (Air)
432-001 (Water)
437-001 (Breath)

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure details the routine for storing and preserving samples at the field sampling site. It includes methods for handling both Tenax (breath, air, and fixed-site) and water samples, and is applicable to any sampling trip/field site.

2.0 SUMMARY OF THE METHOD

All samples are maintained at controlled storage conditions at the field sampling site to ensure the integrity of the samples. Water samples require refrigeration, and Tenax samples require isolation and controlled atmosphere storage.

3.0 PROCEDURE

3.1 Water Samples

3.1.1 Maintain the TEAM field site refrigerator (for samples and ice packs only) at 4°C ± 2°C. Check the temperature daily.

CAUTION

Do not store samples in the refrigerator until the temperature has initially equilibrated. To avoid possible freezing of samples and consequent sample loss, do not store any samples on the shelf directly below the freezer compartment.

3.1.2 Upon arrival at the field sampling site and after equilibration of the refrigerator, remove the Quality Control (code-

QC) samples from the cooler(s) and place them in the refrigerator where they may be readily accessed for transport to/from the collection sites.

NOTE: The QC samples contain sodium thiosulfate as a preservative, as do the 2-oz. bottles that have been readied for sample collection.

3.1.3 After a sample(s) has been collected and returned to the workroom, remove the water bottle(s) from the transport cooler, ensure the security of the cap(s), and place the bottle(s) upright on a shelf in the refrigerator. Do not remove samples from the refrigerator until preparation for transport back to RTI.

3.1.4 For ease of QA checks in shipment preparation and to maintain the temperature of the samples as near to the refrigerated temperature as possible, store samples from different sites on separate shelves (if more than one site is sampled simultaneously).

3.2 Tenax Air and Breath Samples

3.2.1 Set up and maintain the Tedlar storage bags for Tenax samples by passing a helium flow of 50-100 mL/min (Standard Grade, 99.9% purity) through a charcoal filter and into the bag (see Figure 1).

NOTE: (1) Compressed air may be substituted for helium in an emergency situation. (2) A Teflon Y may be inserted in line after the plug valve when one cylinder is used to supply gas to two bags.

3.2.3 Upon initial arrival of Tenax and after equilibrium of the Tedlar bag(s), remove the cans of Tenax cartridges from the footlockers in which they were transported and place cans containing all unused and all QC cartridges in one Tedlar bag.

NOTE: For large studies, more than one Tedlar bag may be required.

3.2.3.1 Remove the four-two inch binder clips, wooden clamp, and dowel from the side of the Tedlar bag that is not heat sealed.

*3.2.3.2 Place approximately 12 cans in each Tedlar bag.

3.2.3.3 Reseal the open side by folding the Tedlar bag once around the dowel (leaving approximately two inches of clearance) replacing the wooden clamp next to the dowel, and securing it with four-two inch binder clips.

3.2.3.4 Separate the female from the male double end shut-off Quick-Connect at the bag inlet. To the female end, attach another male double end shut-off Swagelok® Quick-Connect attached to a 1/4" o.d. piece of Teflon tubing. Attach this tubing to a bubble meter, and assure that the flow to the inlet measures 50-100 mL/min.

3.2.3.5 For each Tedlar bag in use, check the flow rate twice daily, in the morning and evening, after cartridges have either been removed for field sample collection or returned after sampling.

3.2.3.6 When preparing for field sampling, remove only the unused and QC cartridges that are required for that sampling period, and place them in the field collection can. Follow Steps 3.2.3.1 to 3.2.3.3 for opening and resealing the Tedlar storage bag(s).

*3.2.3.7 After exposed cartridges have been returned from the field, store them in cans by matrix and chronological order in one of the Tedlar storage bags as directed by the Site Administrator. Depending on the total number of cans, there may not be room to segregate exposed cartridges in one bag. Follow Steps 3.2.3.1 to 3.2.3.3 for opening and resealing the Tedlar storage bag.

3.2.3.8 Clearly label the cans as to matrix and the date that the first Tenax cartridges were stored in that can.

CAUTION

Do not store more than 17 cartridges per can or else the chance of breakage in transit will be increased.

3.2.3.9 After a can has been filled, label it "full", number the can, and begin a new can for that matrix.

3.2.3.10 Keep the cans of exposed Tenax cartridges in the Tedlar bags under helium atmosphere until they are ready for QA check and subsequent shipment back to RTI.

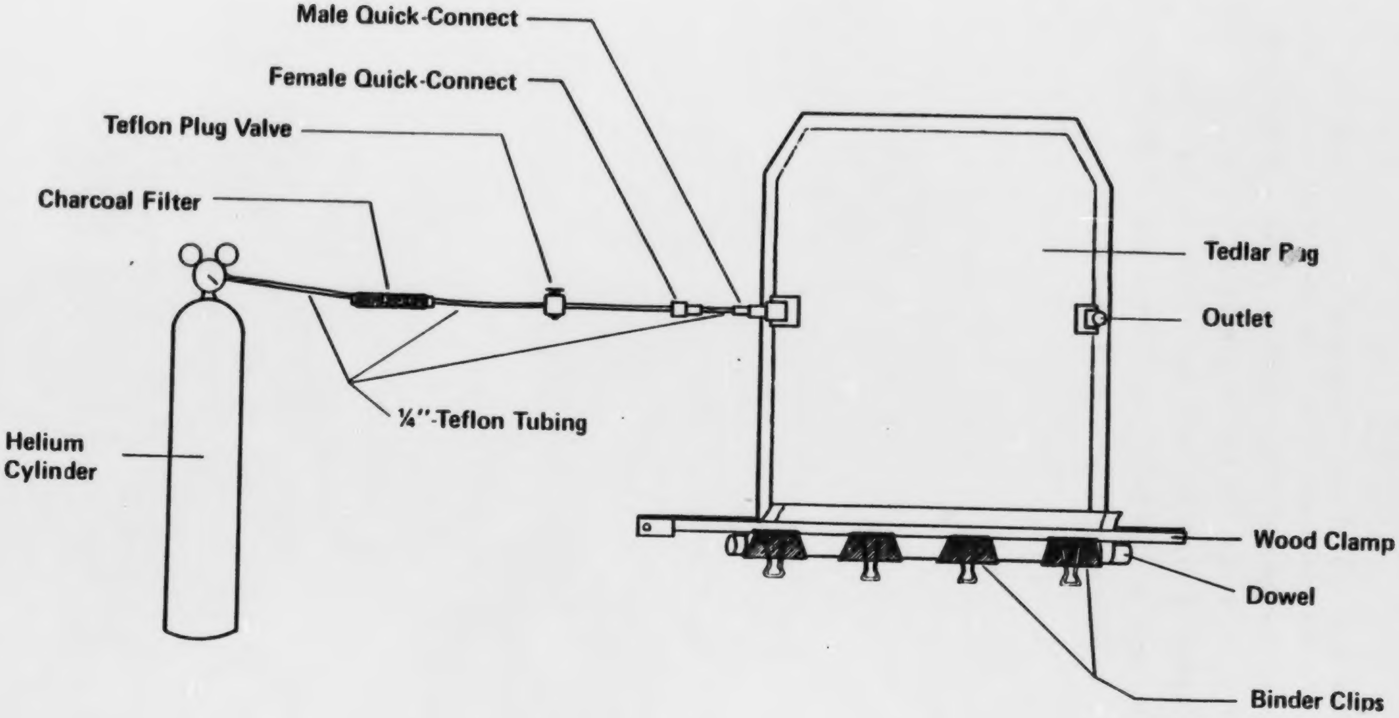
Male Quick-Connect
Female Quick-Connect
Teflon Plug Valve
Charcoal Filter
Helium Cylinder
¼"-Teflon Tubing
Tedlar Bag
Outlet
Wood Clamp
Dowel
Binder Clips

Figure 1. Tedlar storage bag assembly.

RTI/ACS-SOP-461-001 (Air)
SOP-462-001 (Water)
SOP-467-001 (Breath)
December 1983
Revision

Standard Operating Procedure for the Shipment of Samples From the Field Sampling Site to Research Triangle Institute

Author:

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12/7/83
Date

RW Handy
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12/7/83
Date

E Pellizzari
Vice-President, RTI/ACS
12/5/83
Date

Standard Operating Procedure for the Shipment of Samples From the Field Sampling Site to Research Triangle Institute

RTI/ACS-SOP-461-001 (Air)
SOP-462-001 (Water)
SOP-467-001 (Breath)

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure describes the method for shipment of all field samples from the field sampling site to Research Triangle Institute (RTI). Documentation herein includes procedures for (1) checking the samples collected (or quality control [QC] sets used) against Sampling Protocol/Chain-of-Custody (SP/COC) Sheets; (2) documenting the shipment plus receipt and transmittal of the samples on COC records; and (3) proper packing of the samples for transport to RTI. The procedure described below pertains to Tenax and water samples; however, the quality assurance (QA) check, documentation, and packing procedures may be applied to other types of samples collected with only minor modifications.

2.0 SUMMARY OF THE METHOD

All field samples undergo a routine procedure for shipment from the field. Samples are matched to the COC sheets and packed for shipment. The water samples are packed in large Coleman coolers (or equivalent) and transported to RTI along with their corresponding SP/COC sheets. The coolers are transported to RTI in the field sampling van(s) in the case of short term sampling trips or by air for lengthy sampling trips. The Tenax air and breath samples, plus SP/COC sheets, are packed in footlockers and shipped by air, regardless of the duration of the sampling trip.

3.0 PROCEDURE

3.1 Water Samples

3.1.1 Secure the notebook containing the SP/COC sheets for field and QC samples from the Site Administrator.

CAUTION

Ensure that these sheets are in numerical order and have been completed through the field QA check.

3.1.2 Open the TEAM refrigerator and remove one bottle at a time.

3.1.3 Ensure that the screw cap is secure.

3.1.4 Match a SP/COC sheet to the sample and pull that sheet.

3.1.5 Transfer the bottle to a small cooler (six-pack or equivalent size) containing two Blue-Ice® packs.

NOTE: This procedure is carried out most efficiently by two persons.

3.1.6 Continue this procedure until one refrigerator shelf has been emptied of bottles or until all the samples have been checked.

3.1.7 Remove the samples from the cooler and return them to the refrigerator for at least one hour before packing for shipment.

3.1.8 Notify the Site Administrator of any discrepancies between samples and SP/COC sheets.

3.1.9 Locate a large Coleman cooler (or equivalent) and sufficient plastic bubble wrap for packing the water bottles.

3.1.10 Line the bottom and sides of the cooler with a layer of plastic bubble wrap.

3.1.11 Pack the water samples in the cooler.

CAUTION

To minimize loss of analytes in headspace, work quickly to keep temperature of water samples near 4°C.

3.1.11.1 Lay the water samples on their sides in two rows across the length of the cooler (cap end facing the sides of the cooler) with bubble wrap surrounding each bottle so that no bottles touch (see Figure 1).

3.1.11.2 Fill the cooler with layers of bottles and bubble wrap.

3.1.11.3 Place two large or three small frozen Blue-Ice® packs (or equivalent) vertically in the center of the cooler after the first 40-50 bottles have been packed.

CAUTION

To prevent volatilization losses, open the cooler only when samples are actually being packed.

3.1.11.4 Fill all unoccupied spaces in the cooler with bubble wrap so that all bottles are completely immobilized.

3.1.11.5 Put a layer of bubble wrap over the top row of samples. Place two large or three small frozen Blue-Ice® packs (or equivalent) on top of the bottles.

3.1.11.6 If required for a snug fit, add a layer of foam on top, and close the cooler.

3.1.11.7 Enter the following information on each COC sheet.

RELIN-QUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	(Your initials)		(Packing date)	Sent to RTI
(Your initials)				

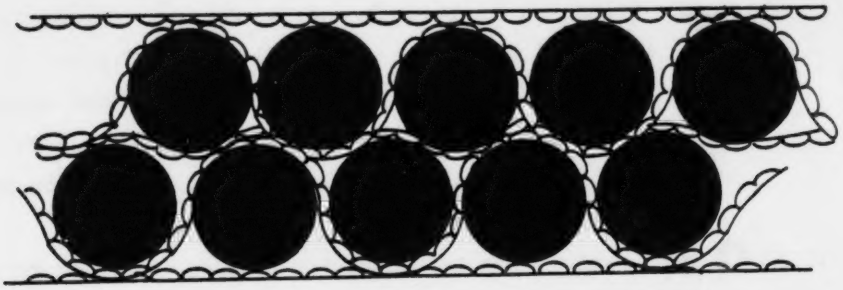


Figure 1. Cross-section of bottles insulated with plastic bubble wrap.

3.1.12 Shipment by Air

3.1.12.1 Put the COC sheets in an envelope and place it inside the cooler.

3.1.12.2 Wrap strapping tape around the cooler on each end to ensure that the cooler does not open during transport.

3.1.12.3 If the cooler is to be shipped to RTI by Federal Express, obtain a Federal Airbill form from the Site Administrator and fill in all pertinent information (see Figure 2).

3.1.12.4 Call the nearest Federal Express office and make arrangements either for pick-up service (if this service is provided in the area) or for delivery to the office by TEAM personnel.

3.1.12.5 If the cooler is to be shipped by a company other than Federal Express, call that company and make the necessary arrangements.

3.1.13 If the samples are to be transported to RTI in the field sampling van, put the SP/COC sheets in a notebook and ensure that this notebook accompanies the samples.

3.2 Tenax Air and Breath Samples

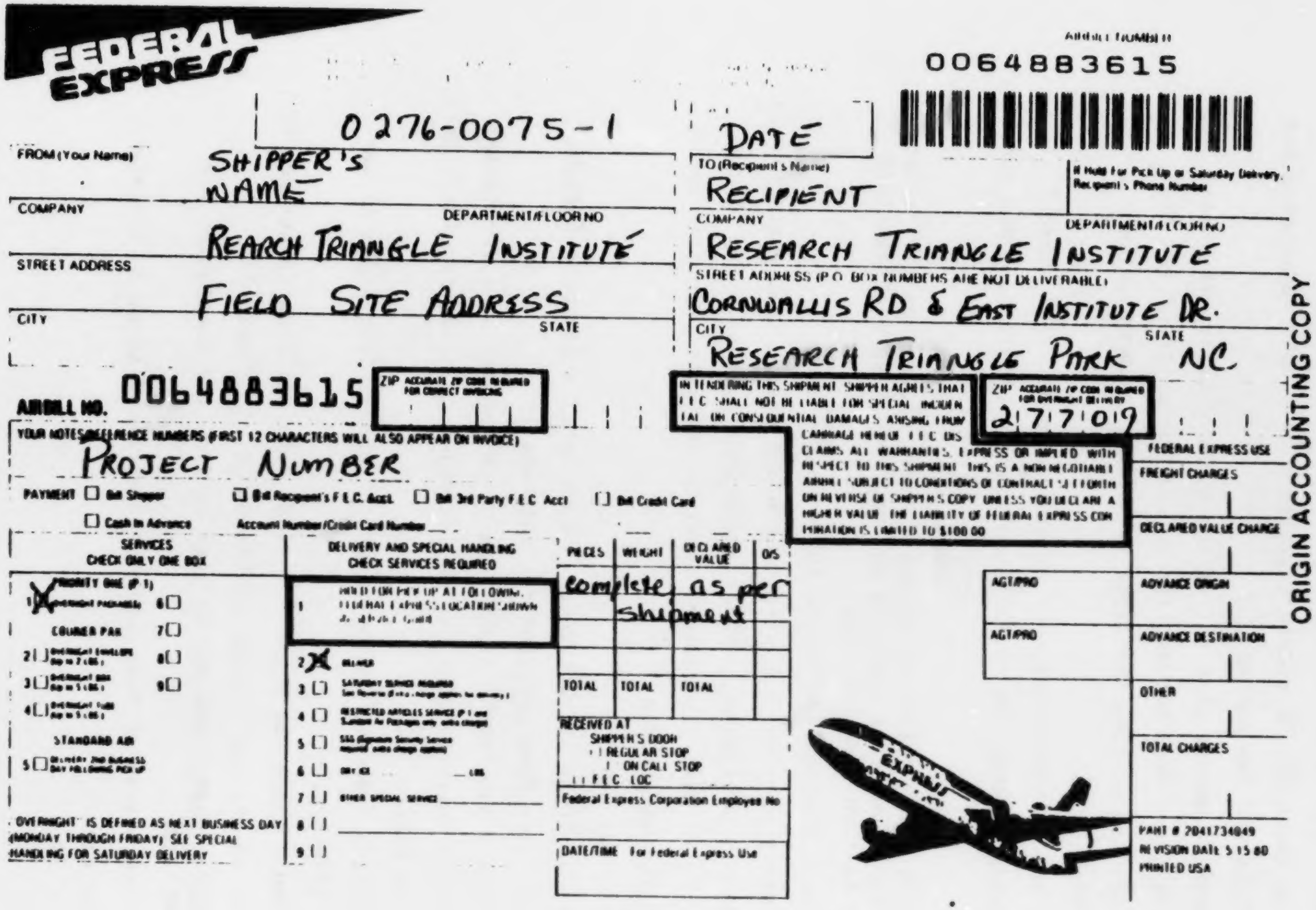
3.2.1 Secure the notebook containing the SP/COC sheets for field and QC samples from the Site Administrator.

CAUTION

Ensure that these sheets are completed through the field QA check.

3.2.2 Remove one can of exposed Tenax cartridges from the Tedlar storage bag.

3.2.3 Open the can, remove the cartridges, and place them on a work table.



FEDERAL EXPRESS

AIRBILL NUMBER
0064883615

0276-0075-1

DATE

FROM (Your Name) SHIPPER'S NAME

COMPANY REARCH TRIANGLE INSTITUTÉ

DEPARTMENT/FLOOR NO

STREET ADDRESS FIELD SITE ADDRESS

CITY

STATE

ZIP ACCURATE ZIP CODE REQUIRED FOR CORRECT INVOICING

AIRBILL NO. 0064883615

YOUR NOTES/REFERENCE NUMBERS (FIRST 12 CHARACTERS WILL ALSO APPEAR ON INVOICE)
PROJECT NUMBER

PAYMENT ☐ Bill Shipper ☐ Bill Recipient's F.E.C. Acct. ☐ Bill 3rd Party F.E.C. Acct ☐ Bill Credit Card

☐ Cash in Advance Account Number/Credit Card Number

TO (Recipient's Name) RECIPIENT

If Hold For Pick Up or Saturday Delivery, Recipient's Phone Number

COMPANY RESEARCH TRIANGLE INSTITUTE

DEPARTMENT/FLOOR NO

STREET ADDRESS (P.O. BOX NUMBERS ARE NOT DELIVERABLE) CORNWALLIS RD & EAST INSTITUTE DR.

CITY RESEARCH TRIANGLE PARK

STATE NC

ZIP ACCURATE ZIP CODE REQUIRED FOR OVERNIGHT DELIVERY 27709

IN TENDERING THIS SHIPMENT SHIPPER AGREES THAT F.E.C. SHALL NOT BE LIABLE FOR SPECIAL INCIDENTAL OR CONSEQUENTIAL DAMAGES ARISING FROM CARRIAGE HEREOF. F.E.C. DISCLAIMS ALL WARRANTIES, EXPRESS OR IMPLIED, WITH RESPECT TO THIS SHIPMENT. THIS IS A NON-NEGOTIABLE AIRBILL SUBJECT TO CONDITIONS OF CONTRACT SET FORTH ON REVERSE OF SHIPPER'S COPY. UNLESS YOU DECLARE A HIGHER VALUE, THE LIABILITY OF FEDERAL EXPRESS CORPORATION IS LIMITED TO $100.00

SERVICES
CHECK ONLY ONE BOX

PRIORITY ONE (P 1)
1 ☒ OVERNIGHT PACKAGES 6 ☐
COURIER PAK 7 ☐
2 ☐ 8 ☐
3 ☐ 9 ☐
4 ☐
STANDARD AIR
5 ☐ DELIVERY 2ND BUSINESS DAY FOLLOWING PICK UP

"OVERNIGHT" IS DEFINED AS NEXT BUSINESS DAY (MONDAY THROUGH FRIDAY). SEE SPECIAL HANDLING FOR SATURDAY DELIVERY

DELIVERY AND SPECIAL HANDLING
CHECK SERVICES REQUIRED

1 HOLD FOR PICK UP AT FOLLOWING FEDERAL EXPRESS LOCATION SHOWN
2 ☒ DELIVER
3 ☐ SATURDAY SERVICE REQUIRED
4 ☐ RESTRICTED ARTICLES SERVICE
5 ☐ SSS (Signature Security Service)
6 ☐ DRY ICE ____ LBS
7 ☐ OTHER SPECIAL SERVICE ________
8 ☐
9 ☐

PIECES	WEIGHT	DECLARED VALUE	O/S
complete	as per		
	shipment		
TOTAL	TOTAL	TOTAL	

RECEIVED AT
SHIPPER'S DOOR
☐ REGULAR STOP
☐ ON CALL STOP
☐ F.E.C. LOC

Federal Express Corporation Employee No

DATE/TIME For Federal Express Use

AGT/PRO

AGT/PRO

FEDERAL EXPRESS USE
FREIGHT CHARGES
DECLARED VALUE CHARGE
ADVANCE ORIGIN
ADVANCE DESTINATION
OTHER
TOTAL CHARGES

PART # 2041734049
REVISION DATE 5 15 80
PRINTED USA

ORIGIN ACCOUNTING COPY

Figure 2. Federal Express Airbill Form.

3.2.4 Match a SP/COC sheet to each sample and notify the Site Administrator of any discrepancies.

3.2.5 Clip these SP/COC sheets together with a note containing the matrix and can number.

3.2.6 Check to ensure that all of the culture tubes are packed with glass wool between the top of the cartrige and the screw cap so that the Tenax cartridge is immobile when shaken.

3.2.7 If any culture tubes need additional glass wool, insert (using forceps) an unsilanized glass wool plug in each tube.

CAUTION

Glass wool protruding from the sides of the cap will reduce effectiveness of the seal and may result in contamination of the Tenax sorbent bed.

3.2.8 Return the sealed culture tubes to the can.

3.2.9 Tightly pack the can with cotton and ensure that each culture tube is surrounded. Place sufficient cotton on top of the tubes so that the culture tubes do not move when the cap is sealed and shaken.

3.2.10 Seal the can.

3.2.11 Repeat Steps 3.2.2 through 3.2.10 until a maximum of eight cans have been prepared for shipment.

3.2.12 Place a maximum of eight cans of cartridges in a footlocker.

3.2.13 Ensure that the cans are well-cushioned inside the footlocker (see Figure 3).

3.2.14 Place a foam pad on the floor and on all four sides of the footlocker.

3.2.15 Evenly space the eight (or less) cans of Tenax cartridges on top of the pad lining the bottom of the footlocker.

NOTE: Empty cans may be used to fill the footlocker, if desired.

3.2.16 Place pieces of foam in between each can so as to isolate the cans.

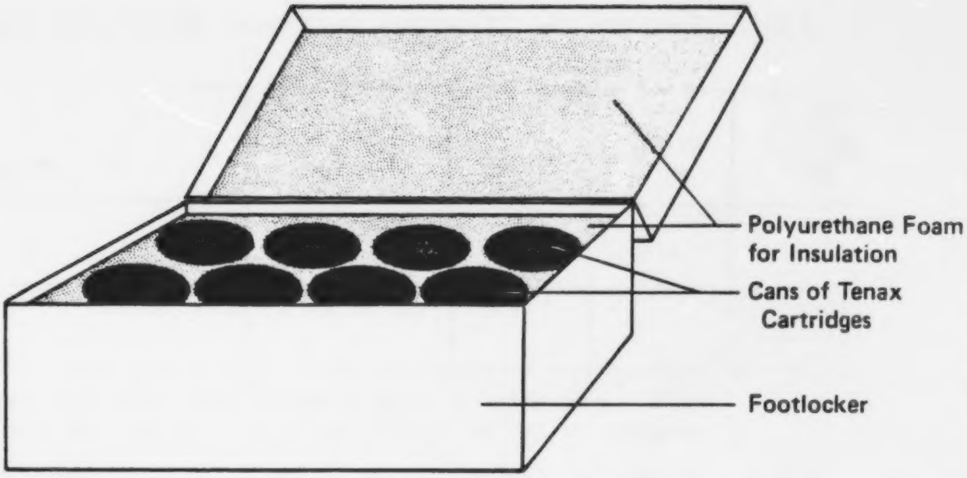

Polyurethane Foam for Insulation
Cans of Tenax Cartridges
Footlocker

Figure 3. Cans of Tenax cartridges cushioned inside a footlocker.

3.2.17 Include the SP/COC sheets in the footlocker after they have been completed (see below).

3.2.18 Place a piece of foam inside the top of the footlocker to secure the cans when the footlocker is closed.

3.2.19 Close and lock the footlocker.

3.2.20 Complete the COC sheets (as stated above) with the following information.

RELINQUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	(Your initials)		(Packing Date)	Sent to RTI
(Your initials)				

NOTE: For ease of logging-in at RTI, keep the COC sheets clipped in groups according to matrix and can number.

3.2.21 If another footlocker is to be readied for shipment, repeat the above procedures beginning with Step 3.2.2.

3.2.22 Wrap strapping tape around both ends of the footlocker(s) to ensure that it does not open during transport.

3.2.23 If the shipment is by Federal Express, obtain a Federal Express Airbill form from the Site Administrator and fill in all pertinent information (see Figure 2).

3.2.24 Call the nearest Federal Express office and make arrangements either for pick-up service (if this service is provided in the area) or for delivery to the office by field personnel.

3.2.25 If the footlocker is to be shipped by a company other than Federal Express, call that company and make the necessary arrangements.

3.1.11.1 Lay the water samples on their sides in two rows across the length of the cooler (cap end facing the sides of the cooler) with bubble wrap surrounding each bottle so that no bottles touch (see Figure 1).

3.1.11.2 Fill the cooler with layers of bottles and bubble wrap.

3.1.11.3 Place two large or three small frozen Blue-Ice® packs (or equivalent) vertically in the center of the cooler after the first 40-50 bottles have been packed.

CAUTION

To prevent volatilization losses, open the cooler only when samples are actually being packed.

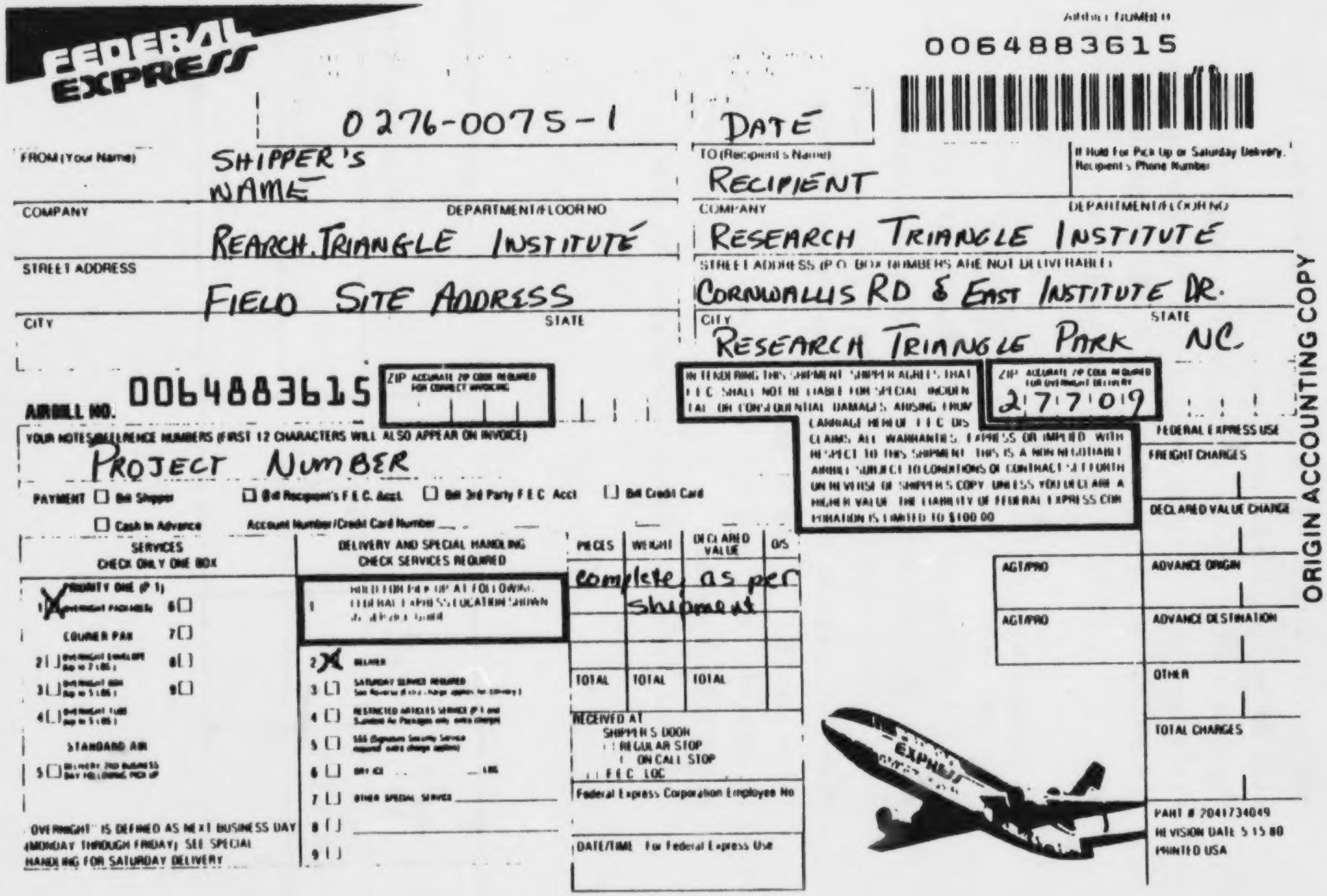
3.1.11.4 Fill all unoccupied spaces in the cooler with bubble wrap so that all bottles are completely immobilized.

3.1.11.5 Put a layer of bubble wrap over the top row of samples. Place two large or three small frozen Blue-Ice® packs (or equivalent) on top of the bottles.

3.1.11.6 If required for a snug fit, add a layer of foam on top, and close the cooler.

3.1.11.7 Enter the following information on each COC sheet.

RELINQUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	(Your initials)		(Packing date)	Sent to RTI
(Your initials)				



FEDERAL EXPRESS

AIRBILL NUMBER
0064883615

0276-0075-1

DATE

FROM (Your Name) SHIPPER'S NAME

COMPANY REARCH. TRIANGLE INSTITUTE
DEPARTMENT/FLOOR NO

STREET ADDRESS FIELD SITE ADDRESS

CITY
STATE

AIRBILL NO. 0064883615

ZIP ACCURATE ZIP CODE REQUIRED FOR CORRECT INVOICING

YOUR NOTES/REFERENCE NUMBERS (FIRST 12 CHARACTERS WILL ALSO APPEAR ON INVOICE) PROJECT NUMBER

PAYMENT ☐ Bill Shipper ☐ Bill Recipient's F.E.C. Acct. ☐ Bill 3rd Party F.E.C. Acct. ☐ Bill Credit Card
☐ Cash in Advance Account Number/Credit Card Number

SERVICES
CHECK ONLY ONE BOX

PRIORITY ONE (P 1)
1 ☒ OVERNIGHT PACKAGES 6 ☐
COURIER PAK 7 ☐
2 ☐ OVERNIGHT ENVELOPE (up to 2 LBS.) 8 ☐
3 ☐ OVERNIGHT BOX (up to 5 LBS.) 9 ☐
4 ☐ OVERNIGHT TUBE (up to 5 LBS.)
STANDARD AIR
5 ☐ DELIVERY 2ND BUSINESS DAY FOLLOWING PICK UP

"OVERNIGHT" IS DEFINED AS NEXT BUSINESS DAY (MONDAY THROUGH FRIDAY). SEE SPECIAL HANDLING FOR SATURDAY DELIVERY

DELIVERY AND SPECIAL HANDLING
CHECK SERVICES REQUIRED

1 HOLD FOR PICK UP AT FOLLOWING FEDERAL EXPRESS LOCATION SHOWN [illegible]
2 ☒ DELIVER
3 ☐ SATURDAY SERVICE REQUIRED See Reverse (Extra charge applies for delivery)
4 ☐ RESTRICTED ARTICLES SERVICE (P 1 and Standard Air Packages only, extra charge)
5 ☐ SSS (Signature Security Service required, extra charge applies)
6 ☐ DRY ICE ___ LBS
7 ☐ OTHER SPECIAL SERVICE ______
8 ☐ ______
9 ☐

PIECES	WEIGHT	DECLARED VALUE	O/S
complete as per shipment			
TOTAL	TOTAL	TOTAL	

RECEIVED AT
☐ SHIPPER'S DOOR
☐ REGULAR STOP
☐ ON CALL STOP
☐ F.E.C. LOC

Federal Express Corporation Employee No

DATE/TIME For Federal Express Use

TO (Recipient's Name) RECIPIENT

If Hold For Pick Up or Saturday Delivery, Recipient's Phone Number

COMPANY RESEARCH TRIANGLE INSTITUTE
DEPARTMENT/FLOOR NO

STREET ADDRESS (P.O. BOX NUMBERS ARE NOT DELIVERABLE) CORNWALLIS RD & EAST INSTITUTE DR.

CITY RESEARCH TRIANGLE PARK
STATE NC

ZIP ACCURATE ZIP CODE REQUIRED FOR OVERNIGHT DELIVERY 27709

IN TENDERING THIS SHIPMENT, SHIPPER AGREES THAT F.E.C. SHALL NOT BE LIABLE FOR SPECIAL, INCIDENTAL OR CONSEQUENTIAL DAMAGES ARISING FROM CARRIAGE HEREOF. F.E.C. DISCLAIMS ALL WARRANTIES, EXPRESS OR IMPLIED, WITH RESPECT TO THIS SHIPMENT. THIS IS A NON-NEGOTIABLE AIRBILL SUBJECT TO CONDITIONS OF CONTRACT SET FORTH ON REVERSE OF SHIPPER'S COPY. UNLESS YOU DECLARE A HIGHER VALUE, THE LIABILITY OF FEDERAL EXPRESS CORPORATION IS LIMITED TO $100.00

AGT/PRO

AGT/PRO

FEDERAL EXPRESS USE
FREIGHT CHARGES
DECLARED VALUE CHARGE
ADVANCE ORIGIN
ADVANCE DESTINATION
OTHER
TOTAL CHARGES

PART # 2041734049
REVISION DATE 5 15 80
PRINTED USA

ORIGIN ACCOUNTING COPY

Figure 2. Federal Express Airbill Form.

3.2.17 Include the SP/COC sheets in the footlocker after they have been completed (see below).

3.2.18 Place a piece of foam inside the top of the footlocker to secure the cans when the footlocker is closed.

3.2.19 Close and lock the footlocker.

3.2.20 Complete the COC sheets (as stated above) with the following information.

RELINQUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	(Your initials)		(Packing Date)	Sent to RTI
(Your initials)				

NOTE: For ease of logging-in at RTI, keep the COC sheets clipped in groups according to matrix and can number.

3.2.21 If another footlocker is to be readied for shipment, repeat the above procedures beginning with Step 3.2.2.

3.2.22 Wrap strapping tape around both ends of the footlocker(s) to ensure that it does not open during transport.

3.2.23 If the shipment is by Federal Express, obtain a Federal Express Airbill form from the Site Administrator and fill in all pertinent information (see Figure 2).

3.2.24 Call the nearest Federal Express office and make arrangements either for pick-up service (if this service is provided in the area) or for delivery to the office by field personnel.

3.2.25 If the footlocker is to be shipped by a company other than Federal Express, call that company and make the necessary arrangements.

RTI/ACS-SOP-470-001
December 1983
Revision 0

Standard Operating Procedure for the Receipt of Air, Breath, and Water Samples at Research Triangle Institute

Author:

Patricia A. Blau
Patricia A. Blau

Approved:

CM Sparacino — 12/7/83
Laboratory Manager, RTI/ACS — Date

12/5/83
QA Officer, RTI/ACS — Date

E Pellizzari — 12/5/83
Vice-President, RTI/ACS — Date

Standard Operating Procedure for the Receipt of Air, Breath, and Water Samples at Research Triangle Institute

RTI/ACS-SOP-470-001

1.0 SCOPE AND APPLICATION

The intention of this Standard Operating Procedure is to describe the step-by-step activities involved in receiving samples at Research Triangle Institute (RTI) from field sampling operations. The methodology herein pertains specifically to air and breath (i.e., Tenax cartridges), and water samples; however, it may be applied to other samples/studies with minor modifications.

2.0 SUMMARY OF THE METHOD

All samples received at RTI, regardless of mode of shipment, shall undergo a routine logging-in procedure. This includes: (1) matching Sampling Protocol/Chain-of-Custody (SP/COC) sheets with samples; (2) attesting the receipt of each sample on the appropriate COC form; (3) inspecting samples for loss and recording such loss; (4) checking SP/COC sheets for completeness; (5) performing a QA check on 15% (minimum) of the calculations; and (6) storage of the samples and distribution of the SP/COC sheets.

3.0 PROCEDURE

3.1 Sample Arrival at RTI

Samples may arrive from the field via air or land transport. The samples may be received by any of the TEAM personnel who will immediately notify the person responsible for logging-in samples. This person shall insure that samples are properly stored prior to (and after) the logging-in procedure. Exposed Tenax cartridges will arrive in sealed paint cans that have been transported in footlockers. Filled water bottles will arrive in Coleman® coolers (or equivalent). The SP/COC sheets will accompany the samples.

3.1.1 Tenax Samples

3.1.1.1 Unpack the footlocker(s).

3.1.1.2 Store the unopened cans in a freezer designated for Tenax cartridges only.

3.1.2 Water Samples

3.1.2.1 Store the cooler(s) in a walk-in refrigerator prior to unpacking.

3.2 Logging-in Samples

3.2.1 Tenax Samples (includes personal and fixed site air and breath samples)

3.2.1.1 Remove one (1) can of Tenax cartridges from the freezer and carry it to the designated logging-in area.

3.2.1.2 Open the can. Remove the cotton packing and save it for future use.

3.2.1.3 Remove the culture tubes containing the Tenax cartridges from the can and place them on the worktable.

3.2.1.4 Locate the SP/COC sheet that matches the first sample and put it in a separate pile that is associated with that can of samples. Verify the consistency of the sample code and batch number.

3.2.1.5 Slip the foil down from the culture tube.

3.2.1.6 Inspect the culture tube for cracks or breakage.

3.2.1.7 Check to insure cap is secure.

3.2.1.8 If the culture tube is damaged, transfer the contents (with forceps) to a clean tube.

3.2.1.9 Inspect the Tenax cartridge for cracks or breakage without opening the culture tube.

3.2.1.10 Reference any damage on the corresponding COC sheet (see 3.2.1.20 below) and note if the culture tube was replaced or cap was loose.

3.2.1.11 Make an entry on the Sample Receipt Report form under "Sample Loss" (see Figure 1).

3.2.1.12 Determine if the damaged cartridge is analyzable.

CAUTION

The cartridge is analyzable if no Tenax has been lost and if the glass is intact around the length of the glass wool plug (i.e., the cartridge can be loaded with PFB/PFT).

3.2.1.13 Separate quality assurance samples from the other samples.

3.2.1.14 If the cartridge is analyzable, return it cap-side-up to the can. Invert the cartridge if it is not analyzable.

NOTE: Inversion of the cartridge is a flag for the person responsible for loading and sending samples to the mass spectrometry facility for analysis.

3.2.1.15 Return all quality assurance samples to a separate can.

3.2.1.16 Repeat Steps 3.2.1.4 through 3.2.1.15 for each sample.

3.2.1.17 Identify the can and its corresponding sheets with any matching label or code (e.g., Can 1, 2, 3 or A, B, C, etc.). Put the code on the lid of the can. Clip the sheets together with a header page containing the code.

NOTE: This coding facilitates locating a particular sample at a later date.

3.2.1.18 Seal the can and return it to the freezer.

3.2.1.19 Remove another can from the freezer and repeat the above procedures beginning with Step 3.2.1.1 until all samples have been examined.

CAUTION

Log-in cans of samples one at a time to minimize the potential for sample contamination.

3.2.1.20 Enter the following information on each COC sheet that has been matched to a sample.

SAMPLE RECEIPT REPORT

Prepared by:
Date:
Sample Trip:

A. Sample Loss

Sample Code	Reason for Loss

B. Other Comments

Figure 1. Sample Receipt Report form (reduced)

RELIN-QUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	(Your initials)		(Logging-in date)	Received at RTI (plus any information on sample loss).
(Your initials)				

3.2.1.21 Scan both sides of each sheet for completeness. Fill in any information from the SP side that is missing on the COC side.

3.2.1.22 Add to the Sample Receipt Report form under "Sample Loss" any samples that were not collected or were lost in the field as documented on the SP/COC sheets by sampling personnel.

3.2.1.23 Obtain a final copy of the Field Sampling Schedule from the Site Administrator.

3.2.1.24 Examine the SP/COC sheets one at a time and check off the respondent numbers on the Schedule. Make sure a sheets exists for each sample that either was or should have been collected.

3.2.1.25 Contact the Site Administrator regarding any discrepancies found. If these discrepancies cannot be resolved, make a note on the Sample Receipt Report form under "Other Comments".

3.2.1.26 Check 15% (minimum) of the sheets for: (1) consistency of front (SP) and back (COC) entries for codes, time(s), temperature(s), volume collected; and (2) elapsed time, flow rate and total volume collected calculations.

3.2.1.27 Document all computational errors found on the Sample Receipt Report form under "Other Comments".

3.2.1.28 Document the total number of noncomputational errors found under "Other Comments".

CAUTION

If more than 5% of the calculations checked contain computational errors made by the same person, then all of the sheets with values calculated by that person must be checked.

3.2.1.29 Make a notation on each COC sheet for which the above QA check has been performed. The entry "QA" may be placed on the same line as "Received at RTI" (see Step 3.2.1.20 above).

3.2.1.30 Make two (2) copies of the completed Sample Receipt Report form and Field Sampling Schedule. Keep the originals in the QA file and give copies to the QA Officer and TEAM liaison to the Computer Applications Center (CAC).

3.2.2 Water Samples

3.2.2.1 Open and unpack the cooler(s) inside the walk-in refrigerator.

3.2.2.2 Put all blanks and controls from a particular city on one tray.

3.2.2.3 Put samples corresponding to the first 20 respondent numbers on another tray, the next 20 respondent numbers on the next tray, etc., until all samples have been removed from the cooler(s) and are on trays.

3.2.2.4 Remove one (1) tray of samples from the walk-in refrigerator and carry it to the designated logging-in area.

3.2.2.5 Remove the samples from the tray and arrange them in numerical order on the worktable.

3.2.2.6 Locate the SP/COC sheet corresponding to the first water sample.

3.2.2.7 Visually inspect the bottle for leakage or breakage. Check to make sure the cap is secure.

3.2.2.8 Reference any damage or loss on the sample's COC sheet (see 3.2.2.13 below) and, if necessary, on the Sample Receipt Report form under "Sample Loss".

3.2.2.9 Repeat Steps 3.2.2.6 through 3.2.2.8 for each sample.

3.2.2.10 Return the samples to the tray in numerical order.

NOTE: This facilitates locating a sample for analysis.

3.2.2.11 Place the tray of samples in the refrigerator designated for water samples.

3.2.2.12 Remove another tray from the walk-in refrigerator and repeat the above procedures, beginning with Step 3.2.2.4 until all samples have been examined.

CAUTION

Log-in one (1) sample at a time to maintain samples near their refrigerated temperature and consequently to minimize partition of analytes into the headspace.

3.2.2.13 Enter the following information on each COC sheet that has been matched to a sample.

RELIN-QUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	(Your initials)		Logging-in date	Received at RTI (plus any information on sample loss).
(Your initials)				

3.2.2.14 Scan both sides of each sheet for completeness. Fill in any missing information.

3.2.2.15 Add to the Sample Receipt Report form under "Sample Loss" any samples that were not collected or lost in the field as documented on the SP/COC sheets for sampling personnel.

3.2.2.16 Obtain a final copy of the Field Sampling Schedule from the Site Administrator. Examine the SP/COC sheets one-by-one, checking off the respondent numbers from the Schedule. Make sure a sheet exists for each sample that either was or should have been collected.

3.2.2.17 Contact the Site Administrator if any discrepancies are found. If these discrepancies cannot be resolved, make an entry on the Sample Receipt Report form under "Other Comments".

3.2.2.18 Check 15% (minimum) of the sheets for consistency of front (SP) and back (COC) entries for codes, times, and volume collected.

3.2.2.19 Make a notation on each COC sheet for which the above QA check has been performed. The entry "QA" may be placed on the same line as "Received at RTI" (see Item 3.2.2.13 above).

3.2.2.20 Document the total number of noncomputational errors found on the Sample Receipt Report form under "Other Comments".

3.2.2.21 Make two (2) copies of the completed Sample Receipt Report form and Field Sampling Schedule. Keep the originals in the QA file and give copies to the QA Officer and TEAM liaison to CAC.

3.3 Handling the SP/COC Sheets

Insure that all SP/COC sheets are in your possession. This includes the sheets for lab blanks and lab controls that remained at RTI during the entire sampling period.

3.3.1 Photocopying

3.3.1.1 For Tenax samples, make one (1) photocopy of each COC record.

NOTE: SP/COC sheets should still be in coded groups according to contents of individual cans.

3.3.1.2 Arrange the Tenax SP/COC sheets in order of ascending responding number for field samples and for blanks and controls (QC samples).

3.3.1.3 Keep SP/COC sheets for field samples separate from QC samples.

3.3.1.4 Make one (1) photocopy of each SP record.

3.3.1.5 For water samples, make sure SP/COC sheets are in ascending order for field samples and QC samples, keeping these two categories separated.

3.3.1.6 Make one (1) photocopy of each COC record and one (1) photocopy of each SP record.

3.3.2 Distribution of SP/COC Sheets

3.3.2.1 Give the COC photocopies corresponding to the samples to be analyzed in-house to the staff member responsible for analysis of the respective samples.

3.3.2.2 Give the COC photocopies corresponding to the QA lab samples to the staff member in charge of QA lab activities.

3.3.2.3 Give the SP photocopies to the Laboratory Manager responsible for that particular matrix.

3.3.2.4 Prepare the originals for binding (see below).

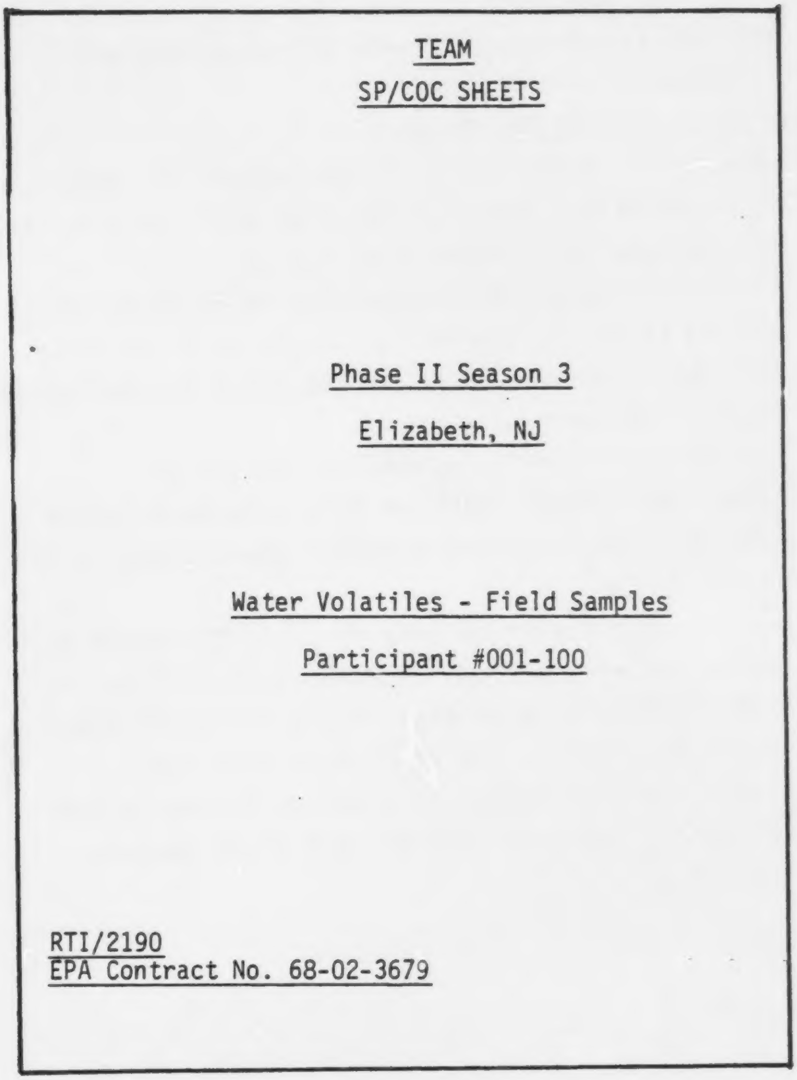
3.3.2.5 Arrange the original, ordered SP/COC sheets by matrix and city, with field sample sheets separate from QC sample sheets.

3.3.2.6 Prepare a cover page for each set of SP/COC sheets to be bound. Include all information depicted in Figure 2.

3.3.2.7 Clip together the cover page and its associated sheets.

3.3.2.8 Number the sheets in the upper right-hand corner.

3.3.2.9 Request the staff member in charge of binding to bind the grouped sheets and deliver them to the Project Director.



TEAM

SP/COC SHEETS

Phase II Season 3

Elizabeth, NJ

Water Volatiles - Field Samples

Participant #001-100

RTI/2190
EPA Contract No. 68-02-3679

Figure 2. Example of a cover page for bound SP/COC Sheets (reduced).

RTI/ACS-SOP-481-001 (Air)
SOP-487-001 (Breath)
December 1983
Revision 0

Standard Operating Procedure for the Storage of Air and Breath Tenax Samples at Research Triangle Institute

Author:

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Approved:

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Laboratory Manager, RTI/ACS Date

RM Handy 12/22/83
QA Officer, RTI/ACS Date

E Pellizzari 12/22/83
Vice-President, RTI/ACS Date

Standard Operating Procedure for the Storage of Air and Breath Tenax Samples at Research Triangle Institute

RTI/ACS-SOP-481-001 (Air)
SOP-487-001 (Breath)

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure describes the proper manner of storing Tenax cartridges on which air and breath volatiles were collected prior to analysis by GC/MS/COMP.

2.0 PRINCIPLE

Several studies have concluded that contamination of Tenax both from external sources and from internal impurities can be minimized by storing the cartridges at -20°C. A freezer reserved for Tenax cartridges is essential.

3.0 PROCEDURE

3.1 Assign a separate shelf of the freezer for each sample type.

3.2 Allocate one shelf for storage of sealed cans containing laboratory Quality Control sets (QC sets), samples to be analyzed by the Quality Assurance laboratory (QA samples) and cartridges loaded with RMR standards awaiting analysis.

NOTE: Upon arrival from the field sampling location, samples are logged-in (see RTI/ACS-SOP-470-001).

3.3 Attest receipt of the sealed cans containing the field samples and store each can in numerical order on the appropriate shelf.

NOTE: Every can is labelled on top with a number indicating the chronological sampling order and the sample matrix.

3.4 Receive the Chain-of-Custody sheets (COC) from the person that logged-in the samples.

NOTE: The COC sheets are organized in coded groups according to the labels on top of each can (i.e., number, sample matrix).

3.5 When the samples are ready to be analyzed, remove the appropriate can from the freezer and allow it to reach room temperature before opening.

3.6 Ensure that a COC sheet exists for each sample in the can.

NOTE: Contact the person in charge of logging-in the samples if any discrepancy is found between the samples and COC sheets.

RTI/ACS-SOP-482-001
December 1983
Revision 0

Standard Operating Procedure for the Storage of Water Samples at the Research Triangle Institute

Author:

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12/7/83
Date

RLHandy
QA Officer, RTI/ACS

12/5/83
Date

E Pellizzari
Vice-President, RTI/ACS

12/5/83
Date

Standard Operating Procedure for the Storage of Water Samples at the Research Triangle Institute

RTI/ACS-SOP-482-001

1.0 SCOPE AND APPLICATION

This SOP describes the method of storing water samples prior to analysis.

2.0 SUMMARY OF THE METHOD

Water samples, including blanks, controls, and field samples are stored at 4°C. Maintaining the samples at this temperature maximizes the percent recovery of the target compounds by retarding partition of the analytes into the headspace.

3.0 PROCEDURE

3.1 Laboratory Blanks and Controls (Lab QC Samples)

3.1.1 Immediately after the lab QC samples have been prepared, place them in the refrigerator designated for TEAM water samples.

NOTE: These samples remain in RTI's TEAM refrigerator until analysis.

3.2 Field Blanks and Controls (Field QC Samples)

3.2.1 Storage Prior to Sampling

3.2.1.1 Immediately after the field QC samples have been prepared, place them in the refrigerator designated for TEAM water samples.

NOTE: These samples are removed from the refrigerator and transported to the field in coolers to maintain refrigeration conditions.

For field storage conditions, see SOP432-001.

3.2.2 Storage at RTI After Sampling

Field blanks and controls are refrigerated in portable coolers during the return to RTI.

3.2.2.1 Upon arrival at RTI, store the coolers containing water samples in the walk-in refrigerator until logging-in has commenced.

3.2.2.2 Following the logging-in procedure (see SOP 470-001) store the samples in the TEAM refrigerator until analysis.

NOTE: The field GC samples are in numerical order on a tray(s) and are removed one at a time immediately prior to analysis.

3.3 Field Samples

3.3.1 Follow Steps 3.2.2.1 and 3.2.2.2 above.

NOTE: These samples are in numerical order on a tray(s) and are removed one at a time immediately prior to analysis.

3.4 QA Samples

3.4.1 These samples remain in the TEAM refrigerator until they are shipped to the QA laboratory.

RTI/ACS-SOP-512-001
December 1983
Revision 0

Standard Operating Procedure for the Analysis of Drinking Water by Purge and Trap/Gas Chromatography

Authors:

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12/16/83
Date

Standard Operating Procedure for the Analysis of Drinking Water by Purge and Trap/Gas Chromatography

RTI/ACS-SOP-512-001

1.0 SUMMARY

Drinking water often contains volatile chemical contaminants such as the trihalomethanes and aromatic hydrocarbons. Some contamination may result from water treatment practices; chlorination often produces chloroform and other chlorinated hydrocarbons. Other contaminants enter the water system as waste products from industrial sites. The identity and quantity of many contaminants are determined by purge and trap/gas chromatography.

Water samples are collected from households, stored in gas tight containers at 4°C, and returned to the laboratory for analysis. A 25 mL aliquot of each sample is analyzed. The aliquot is transferred to a purge flask where a stream of helium is forced through the water. Volatile contaminants partition into the helium and are carried out of the water and onto a trap containing Tenax GC. The contaminants remain adsorbed on the Tenax until the temperature is rapidly raised to 180°C. A nitrogen stream carries the volatile components onto the front of a gas chromatography column. Individual components are separated by the column. The column effluent is split between a flame ionization detector (for detection of aromatic hydrocarbons) and a Hall electrolytic conductivity detector (for detection of halogenated hydrocarbons). Component peak areas are measured and recorded with integrators.

The analytical system is calibrated by analyzing standards prepared in water at several concentrations. Calibration curves are generated for each target compound over the concentration ranges typically found in drinking water. Peak areas obtained during the analysis of samples are compared to the calibration curves for determination of concentration. Analytical performance quality is constantly monitored through the use of system checks, purge blanks, daily performance standards, analysis of blanks and controls, and analysis of blind performance standards.

2.0 MATERIALS AND REAGENTS

2.1 Purge and Trap System

2.1.1 Valco six port high temperature valve.

2.1.2 Purge flask (all glass) for 25 mL samples (Figure 1).

2.1.3 15 mm septum caps for purge flask.

2.1.4 15 mm Teflon-coated rubber septa.

2.1.5 1/4" to 1/8" stainless steel reducing union with nuts and Teflon ferrules on 1/4" end.

2.1.6 1/4" to 1/16" stainless steel reducing union with nuts and Teflon furrules on 1/4" end.

2.1.7 Two 1/4" to 1/16" stainless steel reducing unions with stainless steel nuts and ferrules.

2.1.8 Six stainless steel Valco 1/16" zero dead volume nuts.

2.1.9 Six feet of 1/16" o.d. and 0.02" or 0.04" i.d. nickel tubing.

2.1.10 Eight feet of 1/8" o.d. stainless steel tubing.

2.1.11 Gas flow meter/controller with fittings, Matheson model 7632 with high accuracy needle valve, 1/8" NPT female fittings, and model 600 tube or equivalent.

2.1.12 1/8" ball valve with fittings.

2.1.13 Helium two stage gas regulator, no. 580.

2.1.14 Trap with Tenax GC, 35/60 mesh, (Figure 2).

2.1.15 Charcoal filter for vent outlet.

2.1.16 Heating tape or equivalent for all 1/16" transfer lines and valve (Valco sells an optional valve heater).

2.1.17 Variable transformer(s) to control transfer line voltage.

2.1.18 Control box for sensing and controlling valve and trap temperatures, including meter.

2.1.19 Four thermocouples, two sensing and two controlling, for connection of control box to trap and valve.

2.1.20 Bubble flow meter.

2.1.21 Aspirator with Teflon tubing which will reach bottom of purge flask.

2.1.22 Assorted clamps and hardware for securing flask, trap,

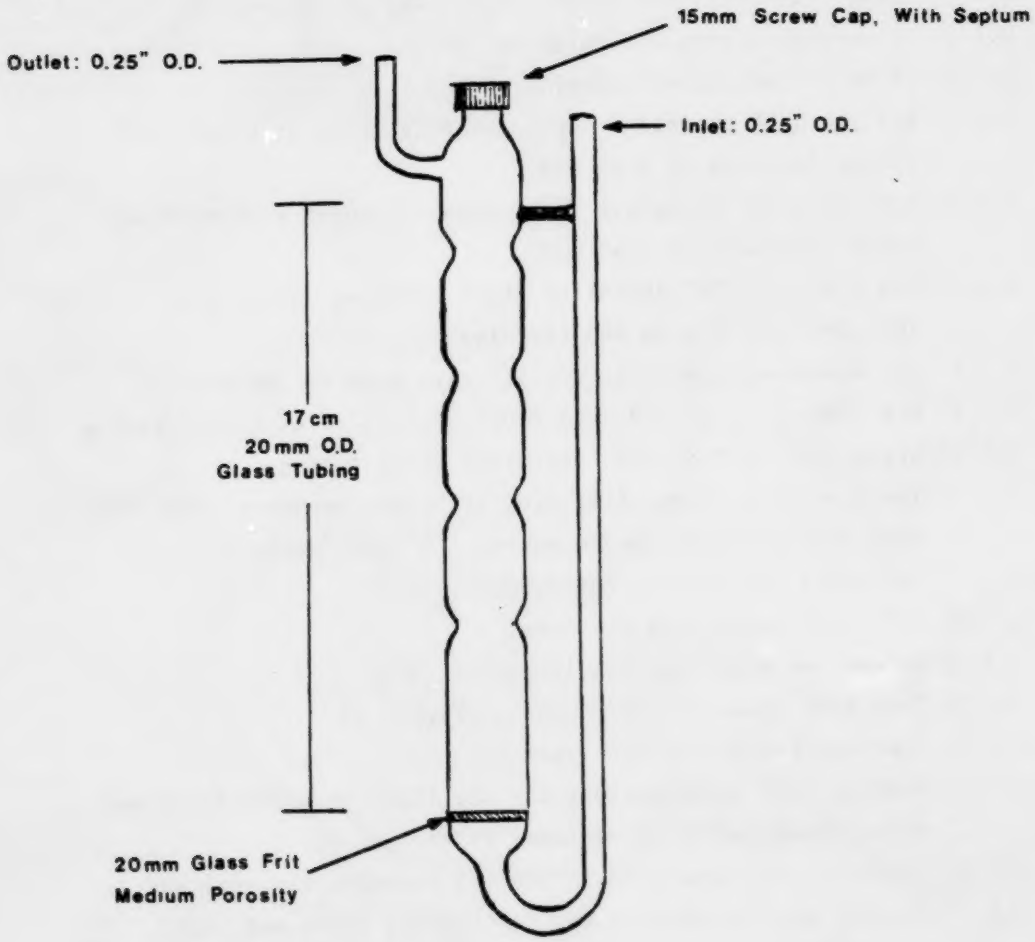
15mm Screw Cap, With Septum
Outlet: 0.25" O.D.
Inlet: 0.25" O.D.
17 cm
20 mm O.D.
Glass Tubing
20mm Glass Frit
Medium Porosity

FIGURE 1. 25 ML PURGE FLASK

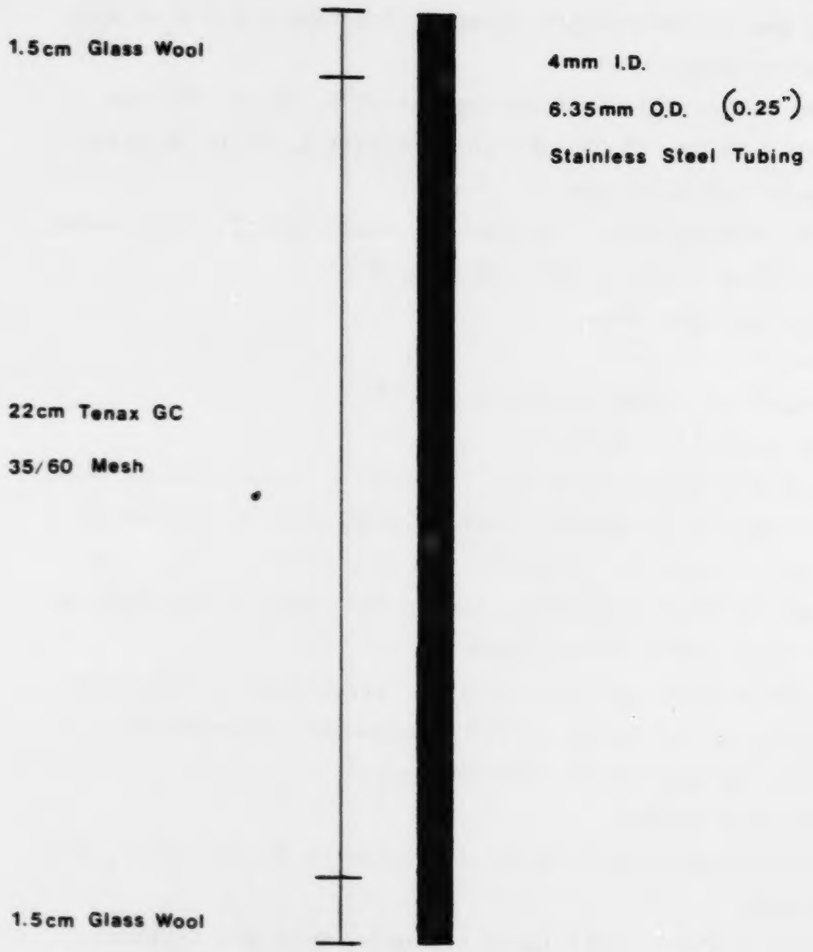
1.5cm Glass Wool
4mm I.D.
6.35mm O.D. (0.25")
Stainless Steel Tubing
22cm Tenax GC
35/60 Mesh
1.5cm Glass Wool

FIGURE 2. TENAX TRAP

valve, charcoal filter, and bubble meter.

2.2 Gas Chromatography System

2.2.1 Gas chromatograph with temperature programming capability which may be interfaced with the purge and trap system.

2.2.2 Glass gas chromatography column 1.7 m long and 2-4 mm i.d.

2.2.3 Silanized glass wool.

2.2.4 Column packing, 5% FFAP on Chromasorb W, 80 to 100 mesh.

2.2.5 Column packing, 1% SP-1000 on Carbopack B, 60 to 80 mesh.

2.2.6 Septa to fit GC column.

2.2.7 Column packing tools: aspirator, small funnel, compressing and picking tools, 5 mL plastic syringe.

2.2.8 Bubble gas flow meter.

2.2.9 Stopwatch.

2.2.10 Nitrogen two stage regulator no. 580.

2.2.11 Flame ionization detector.

2.2.12 Hall electrolytic conductivity detector, model 700 or 700A.

2.2.13 Two integrators, Hewlett Packard model 3390 or equivalent.

2.2.14 Two patch cords for detector to integrator.

2.2.15 Two strip chart recorders, Fisher Recordall Series 5000 or equivalent, with marking pens.

2.2.16 Two patch cords for detectors or integrators to recorders.

2.2.17 Complete set of tools including wrenches, screwdrivers, pliers, tubing cutter, forceps, etc.

2.2.18 Small step ladder.

2.2.19 Nupro stainless steel model 1SG metering valve, 1/16", with fittings.

2.2.20 Stainless steel 1/16" three way union with all fittings.

2.2.21 Teflon tubing, 1/16" i.d., two feet.

2.2.22 Nickel tubing 0.02" i.d., three feet.

2.3 Miscellaneous Equipment

2.3.1 Two 5 µL SGE syringes, plunger in needle, 11.5 cm needles, with plunger guide.

2.3.2 Repair kits for syringes described in 2.3.1.

2.3.3 Two 30 mL gastight syringes, Hamilton no. 1030 or equivalent.

2.3.4 Two 8" x 18 gauge stainless steel needles, Becton-Dickinson No. 6723 or equivalent.

2.3.5 Kimwipes.

2.3.6 Several 150 mL beakers.

2.3.7 A 120 mL Erlenmeyer flask with ground glass joint and stopper.

2.3.8 Several large three ring binder notebooks.

2.3.9 Replacement paper including writing pads, strip chart paper, and integrator print-out paper.

2.3.10 Calculator, Texas Instruments Model 55 or equivalent.

2.4 Reagents

2.4.1 Nitrogen gas, standard grade, 99.995%.

2.4.2 Helium gas, standard grade, 99.995%.

2.4.3 Breathing air, standard dry grade.

2.4.4 Hydrogen gas, standard grade, 99.995%.

2.4.5 Methanol, Burdick and Jackson.

2.4.6 1-Propanol, Burdick and Jackson.

2.4.7 Clean water; distilled or Milli-Q water (deionized and charcoal filtered).

2.4.8 Secondary calibration solutions (see SOP Preparation of Purge and Trap Calibration Solutions).

2.4.9 Spare HECD ion exchange resins.

2.4.10 Dichlorodimethylsilane for silanizing (deactivating) column and HECD reaction tube surfaces.

3.0 INSTRUMENT SET-UP

3.1 Overall Description

A purge and trap unit is a front end sample concentration and delivery system. From a helium gas tank, a tube is run through a gas metering rotometer. From the rotometer, gas flows through the glass frit of the purge flask and strips volatile components from the water. The helium then flows through the Tenax GC trap held at ambient temperature. Next, the six port valve is switched, the trap is heated rapidly to 180°C, and the trap is backflushed with nitrogen gas, sweeping target compounds to the gas chromatography column. Target compounds are separated as the GC column is temperature programmed from 50°C to 220°C. The column effluent is split with five parts to the flame ionization detector and one part to the Hall electrolytic conductivity detector. Peak areas are measured with recording integrators.

3.2 Purge and Trap Unit

The purge and trap unit is built as described below and in Figure 3. Alternatively, a commercial unit such as the Tekmar automated purging unit may be employed with proper modifications and validations.

3.2.1 Construction of Purge and Trap Unit

3.2.1.1 Attach no. 580 regulator to helium gas cylinder.

3.2.1.2 Run a 1/8" tube from cylinder to gas rotometer, and place a three way valve in this line.

3.2.1.3 Run a 1/8" line from rotometer to a 1/8" to 1/4" reducing union.

3.2.1.4 Attach the reducing union to the inlet arm of the purge flask using Teflon ferrules.

3.2.1.5 Attach a 1/4" to 1/16" reducing union to the outlet arm of the purge flask using Teflon ferrules.

3.2.1.6 Run 1/16" o.d. tubing from the reducing union to port F (Figure 3) of valve.

3.2.1.7 Run 1/16" tube from valve port C to the reducing union of Tenax trap.

3.2.1.8 Attach another 1/16" tube from the trap to valve port D.

3.2.1.9 From valve port E run a 1/16" tube to a charcoal filter. This is the vent line.

3.2.1.10 From the valve port B run a 1/16" tube to the front end of the gas chromatography column.

3.2.1.11 Wrap all 1/16" transfer tubes with heating tape except the vent tube..

3.2.1.12 Wrap the six port valve with heating tape (if Valco valve heater is unavailable).

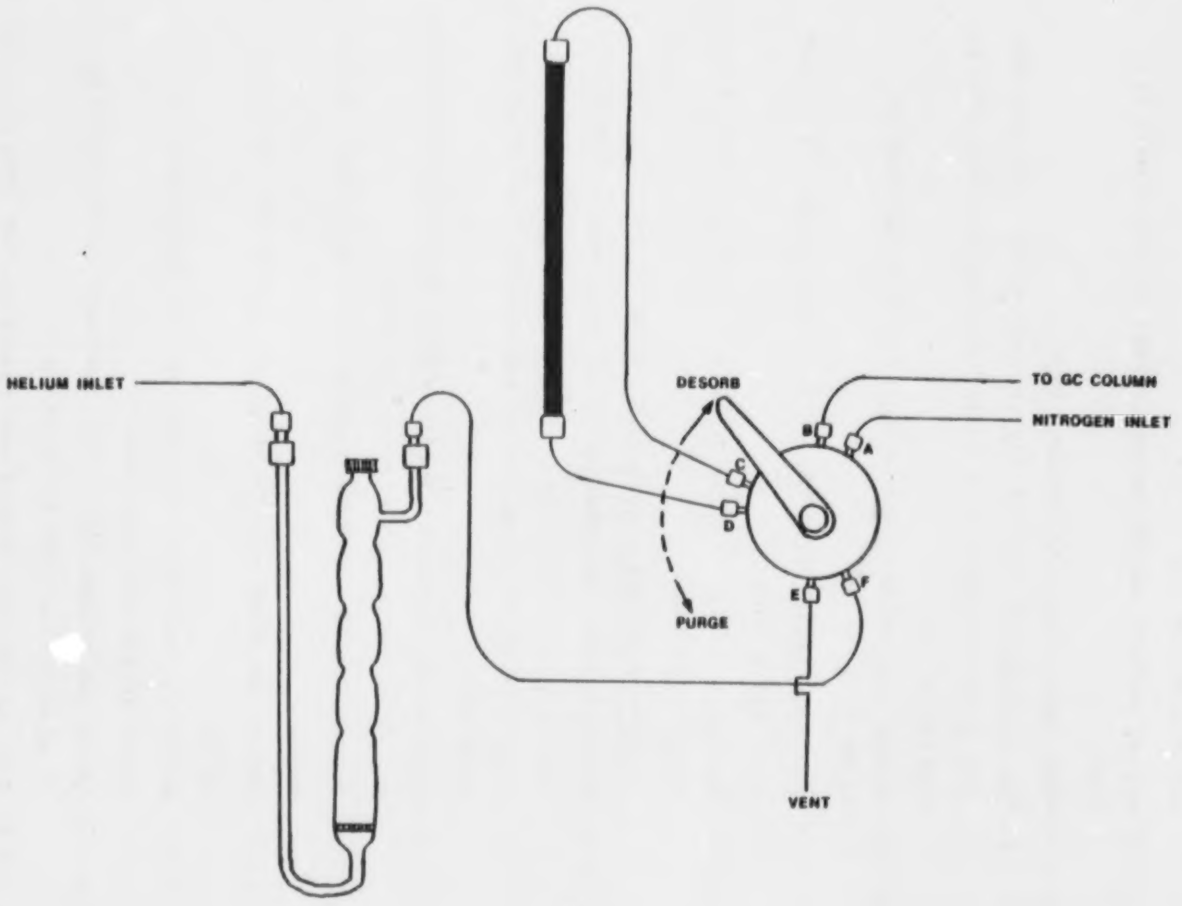
HELIUM INLET
DESORB
PURGE
TO GC COLUMN
NITROGEN INLET
A
B
C
D
E
F
VENT

FIGURE 3. PURGE AND TRAP SYSTEM

3.2.1.13 Connect transfer line heaters to a Variac. Adjust the voltage so lines are maintained at 150°C.

3.2.1.14 Run sensing and controlling thermocouples (with sensing ends glass encased) from under the valve heating tape to the controller box.

3.2.1.15 Adjust voltage output so valve heat is maintained at 150°C.

3.2.1.16 Wrap Tenax trap with heating tape.

3.2.1.17 Run sensing and controlling thermocouples (with sensing ends glass encased) from under the trap heating tape to controller box.

3.2.1.18 Adjust trap voltage output so trap is maintained at 180°C.

3.2.1.19 Run 1/16" tubing from the GC nitrogen gas source to the Valco valve port A.

3.2.1.20 Connect a bubble flow meter to outlet of charcoal filter on the vent line.

3.2.2 Flow Adjustment and Trap Conditioning

3.2.2.1 Adjust helium regulator to 30-40 psig output pressure, with needle valve closed.

3.2.2.2 Adjust nitrogen regulator to approximately 40 psig output pressure.

3.2.2.3 Open helium regulator needle valve, open three way valve, put Valco valve in the purge position.

3.2.2.4 Measure the helium flow rate at the vent bubble flow meter.

3.2.2.5 Adjust the flow rate to 40 mL/min using the rotometer valve.

3.2.2.6 Allow the helium to flow through the Tenax trap for two hours at ambient temperature.

3.2.2.7 Check the helium flow after two hours to be sure the 40 mL/min flow rate is maintained.

3.2.2.8 Turn on the trap heater and condition the trap for four to six hours at 180°C with 40 mL/min helium flow.

CAUTION

The Tenax trap should never be heated unless there is a sufficient helium or nitrogen gas flow to keep air out of the entire system. The purge flask should always be tightly capped.

3.3 Gas Chromatography Unit

3.3.1 Preparation of GC Column

3.3.1.1 Rinse a clean 1.7 m x 4 mm i.d. glass chromatography column with methanol, toluene, and pentane. (Pull solvents through with an aspirator.)

3.3.1.2 With a 5 mL syringe providing suction, draw 1 N NaOH into the column until full. Let sit for five minutes.

3.3.1.3 Empty column, rinse with clean water and the three solvents.

3.3.1.4 In a ventilation hood use a 5 mL syringe to draw 10% dimethyldichlorosilane in toluene until the column is full. Let sit for 30 minutes.

WARNING

Dimethyldichlorosilane is extremely irritating to eyes, nose, and lungs. Do not breath vapors! Use only in ventilation hood.

3.3.1.5 Empty silanizing reagent from column and neutralize waste reagent with methanol.

3.3.1.6 Rinse column with toluene while still in hood.

3.3.1.7 Dry column in oven for at least thirty minutes, preferably two hours.

3.3.1.9 Cool column.

3.3.1.10 Pack silanized glass wool into outlet end of GC column.

3.3.1.11 Apply a vacuum to outlet end of GC column, making sure glass wool does not move.

3.3.1.12 Add portions of 1% SP-1000 on Carbopack B 60/80 mesh to column through a funnel. Rapidly tap the column with a pencil to provide sufficiently tight packing.

3.3.1.13 Leave eight centimeters at the front of the column empty.

3.3.1.14 Add 5 cm of 5% FFAP on Chromasorb W 80/100 mesh packing to front of column.

3.3.1.15 Insert silanized glass wool into front of column.

3.3.2 Installation of Column

3.3.2.1 Place 1/16" transfer line from the purge and trap system through the inlet septum of the chromatography column.

NOTE: When GC systems other than the Fisher Victoreen are used, custom made inlets and outlets must be used for proper gas flow. Inlets must be constructed to connect to the 1/16" line from the purge and trap system. Outlets must be constructed to connect to the effluent splitter.

3.3.2.2 Into the rear septum place a 3 to 4 cm length of 0.04" i.d. 1/16" o.d. nickel or stainless steel tubing.

3.3.2.3 Connect 1/16" i.d. Teflon tubing from the column outlet tube to the short section of 1/16" nickel or stainless tubing which is connected to the stainless steel 1/16" three way union.

3.3.2.4 From the union, one 1/16" tube is connected to the flame ionization detector while the second 1/16" tube is connected to the Nupro fine metering valve.

3.3.2.5 Connect a 1/16" tube from the outlet of the metering valve to the combustion tube of the HECD.

NOTE: In order to achieve the 5-1 split ratio between FID and HECD, the outlet tube positions from the splitter to detectors may be switched to compensate for back pressure differences.

3.4 Detectors

3.4.1 Flame Ionization Detector

3.4.1.1 Clean FID (if necessary) according to GC manual instructions.

3.4.1.2 Connect hydrogen gas line to cylinder regulator and FID.

WARNING

Hydrogen is a highly flammable gas, handle with care.

3.4.1.3 Connect air gas tube to cylinder regulator and FID.

3.4.1.4 Connect column effluent line from splitter to FID.

3.4.2 Hall Electrolytic Conductivity Detector

3.4.2.1 Attach HECD furnace to the GC as close to the splitter as possible.

3.4.2.2 Install quartz combustion tube as described in HECD manual.

3.4.2.3 Connect hydrogen line from cylinder to combustion tube union.

3.4.2.4 Connect column effluent line from splitter to the HECD combustion tube.

3.4.2.5 Load ion exchange resin to HECD as instructed in manual.

3.4.2.6 Fill HECD reservoir with 1:1 1-propanol and Milli-Q or distilled water.

3.4.2.7 Connect all Teflon lines according to HECD manual.

3.4.2.8 Adjust the HECD furnace temperature to 900°C.

3.5 Flow Checks and Conditioning

3.5.1 Nitrogen Carrier Gas

3.5.1.1 Place Valco valve in desorb position.

3.5.1.2 Adjust nitrogen cylinder regulator to 40 psig output.

3.5.1.3 Attach a bubble flow meter to the metal 1/16" column outlet tube.

3.5.1.4 Check for leaks in the carrier gas line from the cylinder to column outlet. Stop all leaks.

3.5.1.5 Adjust column flow to 30 mL/min with the GC rotometer valve.

3.5.1.6 Remove bubble flow meter from column.

3.5.1.7 Do not connect column to detectors until the column has been conditioned.

3.5.2 GC Column Conditioning

3.5.2.1 Allow nitrogen to flow through GC column for at least one hour at 50°C.

3.5.2.2 Start a temperature program of 1°C/min from 50°C to 220°C. Hold at 220°C overnight.

3.5.3 Flow Rate Adjustment

3.5.3.1 Cool column to 50°C and allow to equilibrate.

3.5.3.2 Measure the column flow rate at the end of the column.

3.5.3.3 Readjust the flow rate to 30 mL/min if necessary.

3.5.3.4 Connect the column outlet to the splitter assembly with 1/16" Teflon tubing.

3.5.3.5 Check for leaks on entire system. Stop any leaks found.

NOTE: Leaks around the Teflon tubing may be stopped by heating tubing with a hot blow gun.

3.5.3.6 Turn on HECD furnace. Allow to equilibrate at 900°C.

3.5.3.7 Measure the column effluent flow rate to the HECD at the Teflon tube leading to the conductivity cell.

3.5.3.8 Start hydrogen flow through HECD combustion tube.

3.5.3.9 Adjust HECD hydrogen flow to approximately 35 mL/min. Measure the combined nitrogen and hydrogen flow rates through the HECD.

3.5.3.10 Reconnect Teflon tube to conductivity cell.

3.5.3.11 Calculate column effluent split to the FID by the equation:

Column Flow (Total) - Effluent Flow (HECD) =
Effluent Flow (FID)

3.5.3.12 Turn on FID reaction gases hydrogen and air. The hydrogen should flow at 30 mL/min and air at approximately 300 mL/min.

NOTE: Check HECD hydrogen flow and readjust if necessary. This is important if the two detectors use the same hydrogen tank.

3.5.4 HECD Solvent Flow

3.5.4.1 Make sure the HECD reservoir is filled with 1:1 1-propanol and water.

3.5.4.2 Turn HECD pump on, wait five minutes.

3.5.4.3 Place the Teflon return hose into a 5 mL graduated cylinder.

3.5.4.4 Allow solvent to flow for eight minutes, then read volume delivered.

3.5.4.5 Adjust solvent flow rate to 0.25 mL/min with metering valve on the back of HECD.

NOTE: The HECD solvent flow rate may be adjusted to obtain a desired sensitivity. However, when calibration curves are generated at a given flow rate, the rate must not be changed for sample analysis.

3.5.4.6 Replace Teflon return tube to reservoir cap.

3.6 Integrators and Recorders

3.6.1 Integrators

3.6.1.1 Attach one end of a patch cord to the HECD computer output terminals.

3.6.1.2 Attach the other end to HP 3390 integrator.

3.6.1.3 Attach one end of a patch cord to the output terminals of the FID electrometer.

3.6.1.4 Attach the other end of the patch cord to a HP 3390 or Spectra Physics System One integrator.

3.6.2 Recorders

3.6.2.1 Strip chart recorders are optional if HP 3390 integrators are used. If integrators which do not plot chromatograms are used, then strip chart recorders are required.

3.6.2.2 Attach one end of a patch cord to the HECD recorder output terminals.

3.6.2.3 Attach the patch cord to the strip chart recorder.

3.6.2.4 Attach one end of a patch cord to the FID electrometer recorder output (or the Spectra Physics integrator recorder output) terminals.

3.6.2.5 Attach the patch cord to the strip chart recorder.

3.7 Purge and Trap/Gas Chromatographic Conditions

The conditions for purge and trap/GC analysis of water samples, controls, blanks, and standard calibration solutions are listed in Table 1.

4.0 INSTRUMENT CALIBRATION

Before samples can be analyzed using the purge and trap/GC system, the system must be calibrated to ensure valid identification and quantitation of the target compounds. The system calibration will involve retention time determinations and the preparation and analysis of calibration standards.

Table 1. PURGE AND TRAP/GC CONDITIONS FOR THE ANALYSIS OF WATER SAMPLES, BLANKS, CONTROLS, AND STANDARDS.

Purge and Trap:	
Sample volume:	25 mL
Helium purge rate:	40 mL/min
Purge time:	12 min.
Initial trap temperature:	<35°C
Final trap temperature:	180°C
Valve temperature:	150°C
Transfer line temperatures:	150°C
Gas Chromatography	
Nitrogen carrier rate:	30-35 mL/min
Injector temperature:	230°C
FID temperature:	260°C
HECD temperature:	900°C
Transfer line to HECD:	200°C
HECD hydrogen rate:	~35 mL/min
HECD solvent flow rate:	~0.3 mL/min
FID hydrogen rate:	30 mL/min
FID air rate:	300 mL/min
Column effluent split ratio: (FID:HECD)	5:1
Temperature program:	60°C for 2 minutes 60°C to 220°C at 6°C/min Hold at 220°C for last peak

4.1 Retention Time Determinations

4.1.1 Prepare solutions of individual compounds in methanol.

NOTE: The exact concentration is not important; however, it should be approximately 10 ng/μL.

4.1.2 Individually analyze each of the solutions following the instructions given in section 6.0 on Sample Analysis.

4.1.3 Determine the retention times and elution order from the integrator reports.

4.2 Calibration Curves

4.2.1 Prepare the calibration standards.

4.2.1.1 Refer to the Standard Operating Procedure on the Preparation of Purge and Trap Calibration Solutions (RTI/ACS-SOP-612-001).

4.2.2 Inject standards as described in Section 6.1.4.

NOTE: The purged water in the purge flask to which the standards are added should be aspirated from the flask and replaced with fresh water after each standard analyzed. This reduces the possibility of problems due to carryover.

4.2.3 Record the area counts on a calibration curve sheet (Figure 4) and put in Sample Log notebook.

5.0 SYSTEM MAINTENANCE

5.1 Gas Chromatograph

5.1.1 Frequently check gas tanks and replace when the gauge indicates approximately 500 psi.

5.1.2 Replace oxy-traps after approximately four gas cylinder changes.

NOTE: It is important that the carrier gas (N_2) be scrubbed of any oxygen since the GC column can be degraded by oxygen.

5.2 Detectors

5.2.1 Periodically measure the solvent flow through the HECD cell.

NOTE: This need only be done about once every week unless a noticable change in sensitivity occurs for no apparent reason.

Calibration Curve Data

Date: ______

Area response is $\times 10^4$

	ppb in 25 mL of water												
	Blank	G	F	E	D	C	B	A	AA				
Compound	0	.05	0.1	0.25	0.5	1.0	2.0	5.0	7.5		slope	Corr.	y-inter.
BEN													
Tol													
Ethylbenzene													
Sty													
p-xylene													
VDC													
DCE													
MC													
CCl_4													
TCE													
CDM													
$CHBr_3$													
Perc													
ClBen													
o-Cl_2Ben													
	0	1.5	5	10	20	50	75	100	152				
$CHCl_3$													
	0	0.3	1.0	2.0	4.0	10	15	20	30				
BDM													

① All FID areas are obtained by multiplying attenuation x counts

② conc. = x
area = y

conc. unk. = $\frac{\text{AREA}}{\text{SLOPE}}$

Figure 4. Calibration curve data form.

5.2.1.1 Disconnect the Teflon tubing leading to the conductivity cell.

5.2.1.2 Place the tubing in a 5 mL graduated cylinder and measure the length of time required to collect 0.50 mL.

5.2.1.3 Calculate the flow rate in mL/min.

NOTE: The flow rate should be in the range of 0.20 to 0.25 mL/min. See the Instrument Log for the previous measurement.

5.2.1.4 Replace the Teflon line to the conductivity cell.

5.2.1.5 Visually check for solvent leaks around connection.

5.2.2 Periodically measure the gas flows to the detectors.

NOTE: It is important that all gas and solvent flows be measured and adjusted before use when the instrument has been idle for any extended length of time.

5.2.2.1 Refer to the Instrument Log notebook for details on measuring and adjusting the gas flows to the detectors.

5.2.3 Clean the detectors following directions found in the operation manuals whenever the background rises above acceptable levels.

5.2.4 Replace the ion exchange resins in the HECD at approximately 3-month intervals.

5.2.4.1 Refer to the HECD Operation Manual for instructions.

5.2.5 Add HECD solvent (1:1 n-propanol/water) to the solvent reservoir.

CAUTION

The solvent should be maintained at a level of one-half to one inch below the top of the reservoir. If the reservoir should ever become dry, irreversible damage may occur to the pump.

6.0 SAMPLE ANALYSIS

6.1 Daily Routine

The steps outlined in this section should be performed each day the system is to be used. Included in the daily routine are the following: system start-up, system check, purge blank, daily standard, sample analyses and shut-down. The system start-up involves the activation of the GC, turning on the transfer line heaters, beginning an off-line water purge and the review and updating of integrator parameters. The system check provides an indication of the background from the GC and readies the GC column for injections. The purge blank verifies the cleanliness of the water into which the daily standard will be spiked. The daily standard is the most important of the daily routine steps. It provides the basis on which to judge the sensitivity and reproducibility of the instrument. By comparing the current day's run to the previous day's run, decisions are made as to whether samples should be analyzed or not. The system shut-down is simply the things to do at the end of the day for overnight or weekend periods of latency.

6.1.1 Begin the system start-up.

6.1.1.1 Turn the Valco valve heater on.

6.1.1.2 Turn the transfer line heaters on.

6.1.1.3 Add 25 mL of Milli-Q water to the purge flask.

NOTE: This should be done by unscrewing the cap and adding the water directly through the purge flask opening. This avoids piercing the septum and reduces the possibility of leaks occurring at the septum.

6.1.1.4 Turn the Valco valve to the "desorb" position.

NOTE: When the valve is in the "desorb" position, the Tenax trap is bypassed. Anything purged from the water will vent through a charcoal trap to the room. This is considered purging "off-line."

6.1.1.5 Turn the ball valve on the helium inlet to the "on" position and begin purging the water.

6.1.1.6 Turn the recorders on and adjust the chart speed to 0.25 cm/min.

6.1.1.7 Turn on the air and hydrogen gases to the FID and light it.

6.1.1.8 Turn the HECD on and adjust the attenuation and conductivity.

6.1.1.9 List the integrator parameters and change if necessary.

6.1.2 Perform the system check.

6.1.2.1 Turn the Tenax trap heater on.

6.1.2.2 Verify that the Valco valve is in the "desorb" position.

6.1.2.3 Initiate the GC program by depressing the start button.

6.1.2.4 Start the integrators and recorders.

6.1.2.5 Turn the Tenax trap heater off after 8-12 minutes.

NOTE: Turning the trap heat off too early may not allow complete desorption from the trap. Turning the trap heat off after too long a time period may lengthen the recycle time since the trap must cool to ambient temperature before another sample can be purged.

6.1.3 Run the purge blank.

NOTE: The purge blank is run immediately after the system check. The water in the purge flask is the water that was added during the system startup.

6.1.3.1 Verify that the Tenax trap is at ambient temperature.

6.1.3.2 Simultaneously rotate the Valco valve to the "purge" position and start the timer.

6.1.3.3 Purge the pre-purged water for 12.0 minutes.

6.1.3.4 At the end of the 12.0 minute purge period, simultaneously rotate the Valco valve to the "desorb" position and turn the trap heater on.

6.1.3.5 Initiate the GC program by depressing the start button.

6.1.3.6 Initiate the integrators for each detector.

6.1.3.7 Start the recorders.

NOTE: Steps 6.1.3.4 to 6.1.3.7 should be performed in the same manner and as reproducibly as possible each time. Failure to do this can result in poor reproducibility of retention times.

6.1.4 Analyze the daily standard.

NOTE: Generally only one daily standard is analyzed each day unless the analysis period exceeds 10 hours. In the case where the instrument is operated continuously over two shifts, a second daily standard should be analyzed at the beginning of the second shift. However, this needs only to be done approximately 40% of the time (i.e., 2 days per 5 days of analysis).

6.1.4.1 Turn the helium ball valve to the "off" position and wait for the rotameter to indicate zero flow.

6.1.4.2 Verify that the Tenax trap is at ambient temperature.

NOTE: If the Tenax trap is not at ambient temperature, wait until it is before proceeding.

6.1.4.3 Rotate the Valco valve to the "purge" position.

6.1.4.4 Inject 5.0 μL of standard B into the purge flask using a 5 μL SGE syringe.

NOTE: Rinse the syringe a minimum of three times with clean methanol and twice with the standard to be injected. The standard should be at room temperature. Slowly fill the syringe with the standard and carefully insert the needle into the septum of the purge flask. Smoothly and cleanly eject the 5 μL of standard under the surface of the pre-purged water. Failure to do the above properly can result in contamination and/or poor reproducibility.

6.1.4.5 Place a drop of water on the pierced septum of the purge flask.

NOTE: This drop of water is to aid in the detection of leaks during the purge period.

6.1.4.6 Turn the helium ball valve to the "on" position.

6.1.4.7 Start the timer.

6.1.4.8 Inspect the drop of water (see step 6.1.4.5) for bubbles.

CAUTION

> The appearance of bubbles in the drop of water on the septum indicates a leak. When this occurs, immediately turn the helium flow off and try to reseal the septum by prodding the leaking area. If sealed quickly the loss will be insignificant. If the septum does not reseal quickly, proceed to step 6.1.4.10, replace the septum and repeat the daily standard.

6.1.4.9 Purge for 12.0 minutes.

6.1.4.10 At the end of the purge period, simultaneously rotate the Valco valve to the "desorb" position and turn the trap heater on.

6.1.4.11 Initiate the GC program by depressing the start button.

6.1.4.12 Initiate the integrators for each detector.

6.1.4.13 Start the recorders.

> NOTE: Steps 6.1.4.10-6.1.4.13 should be performed in the same manner and as reproducibly as possible each time. Failure to do this can result in poor reproducibility of retention times.

6.1.4.14 Turn the Tenax trap heater off after 8-12 minutes.

6.1.4.15 Aspirate the water from the purge flask.

6.1.5 Begin sample analysis.

6.1.5.1 Follow instructions in Section 6.2.

6.1.6 System shut-down.

6.1.6.1 Turn the helium tank valve and ball valve off.

6.1.6.2 Rotate Valco valve to the "desorb" position.

6.1.6.3 Turn off the transfer line heaters.

6.1.6.4 Turn off the valve heater.

6.1.6.5 Switch the GC to the "standby" mode.

6.1.6.6 Turn the FID electrometer off.

6.1.6.7 Turn the HECD detector off.

6.1.6.8 Turn the recorders off.

6.2 Purging of Water Samples

6.2.1 Determine the sample to be analyzed.

NOTE: Samples are analyzed according to the collection date. The oldest samples are analyzed first.

6.2.2 Turn the helium ball valve to the "off" position and wait for the rotameter to indicate zero flow.

6.2.3 Verify that the trap is at ambient temperature.

NOTE: If the temperature of the Tenax trap is significantly above ambient, breakthrough volumes for some compounds can be exceeded. For this reason, never begin purging a water sample until the trap temperature reaches room temperature.

6.2.4 Rotate the Valco valve to the "purge" position.

6.2.5 Transfer 25.0 mL of the sample to the purge flask using a 30 mL gastight syringe.

6.2.5.1 Remove the water sample from the refrigerator.

CAUTION

The sample should not be removed from the refrigerator or opened until immediately prior to filling the syringe. Transfer the sample while cold to minimize any loss of volatile compounds.

6.2.5.2 Unscrew the cap and immediately withdraw approximately 5 mL of sample into the syringe; rinse and eject.

6.2.5.3 Repeat 6.2.5.2 once more.

6.2.5.4 Slowly fill the syringe with approximately 30 mL of sample being careful to keep the headspace to a minimum.

6.2.5.5 Invert the syringe with the needle pointing up and gently tap the bubbles to the top.

6.2.5.6 Eject the headspace and adjust the volume to 25.0 mL.

6.2.5.7 Pierce the septum of the purge flask and inject the 25.0 mL of water.

6.2.6 Place a drop of water on the pierced septum.

NOTE: This is to aid in the detection of leaks in the septum during the purge period.

6.2.7 Turn the helium ball valve to the "on" position.

6.2.8 Start the timer.

6.2.9 Inspect the drop of water (see 6.2.6) for bubbles.

CAUTION

The appearance of bubbles in the drop of water on the septum indicates a leak. When this occurs, immediately turn the helium flow off and try to reseal the septum by prodding the leaking area. If the septum does not reseal, as a last resort the septum can be quickly changed after the helium flow has stopped. Make appropriate notations in the sample log and on the COC sheet. Turn the helium on and continue the purge.

6.2.10 Purge for 12.0 minutes.

6.2.11 Check and adjust the HECD and FID attenuations during the purge period if necessary.

6.2.12 After 12.0 minutes, simultaneously rotate the Valco valve to the "desorb" position and turn the trap heater on.

6.2.13 Initiate the GC program by depressing the start button.

6.2.14 Initiate the integrators for each detector.

6.2.15 Start the recorders.

NOTE: It is important that the above steps (6.2.12-6.2.15) be performed in the same order and as reproducibly as possible each time. Failure to do this can result in poor reproducibility of retention times.

6.2.16 Turn the Tenax trap heat off 8-12 minutes after initiation.

6.2.17 Rinse the syringe with organic-free water.

6.2.18 Aspirate the purged sample from the purge flask.

6.2.19 Continue to purge the purge flask with helium in the off-line position until ready to add the next sample.

6.3 Blanks and Controls

Analyses of field blanks and controls and lab blanks and controls are performed following the same procedure as samples. The blanks and controls are stored and analyzed interspersed among the water samples. The frequency of analysis for blanks and controls is generally one set (either one lab blank and control or one field blank and control) for every 6-8 samples. A blank and its corresponding control should be analyzed one after the other and both on the same day. It is preferable to analyze the blank first to minimize carryover from the control to the blank. Choice of the lab blank and control set to be analyzed is determined by its numerical order. For example: a LB-1 and LC-1 would be analyzed before a LB-2 and LC-2. Field control sets are analyzed according to the date they were exposed in the field. Normally, this is also in numerical order.

6.4 Calibration Standards

Quantitation of the water samples, blanks and controls is accomplished using eight calibration standards and a methanol blank. These standards supply the necessary data points to construct a calibration curve. Two complete sets of calibration standards must be satisfactorily analyzed before any samples can be analyzed. This provides an indication of the systems performance prior to starting the samples. After the initial sets of calibration standards are run, additional sets should be run at intervals of approximately once every 10 analysis days. Once all samples have been analyzed, one final calibration set is run. If any unexpected problems are encountered with the system or any alterations or maintainence performed that might affect the instrument response, a set of calibration standards must be analyzed.

Analysis of the set of calibration standards requires one day to complete. The routine is very similar to the daily routine described in Section 6.1. The major differences are that 5 μL of the methanol used in the standards is added to the purge blank and the daily standard is replaced with the lowest concentration standard. The remainder of the standards are then analyzed in order of increasing concentration. Injections are made as explained in Section 6.1.4. The purge water is changed after each standard and purged off-line for approximately 40 minutes during the GC program.

6.4.1 Run the calibration curve.

6.4.1.1 Begin system start-up.

6.4.1.2 Run the system check.

6.4.1.3 Run the methanol blank.

6.4.1.4 Analyze calibration standard G.

6.4.1.5 Analyze calibration standard F.

6.4.1.6 Analyze calibration standard E.

6.4.1.7 Analyze calibration standard D.

6.4.1.8 Analyze calibration standard C.

6.4.1.9 Analyze calibration standard B.

6.4.1.10 Analyze calibration standard A.

6.4.1.11 Analyze calibration standard AA.

7.0 DATA HANDLING

The primary considerations in this section are the storage of the chromatograms, charts, integrator reports and the general interpretations of system checks, daily standards and calibration curves. For quantitation procedures see the Standard Operating procedure titled Quantitation of Team Target Compounds.

7.1 Labeling

All chromatograms and integrator reports must be properly labeled to aid in easy reference and to minimize mistakes. Chromatograms should be labeled with the date analyzed, the operators initials, the sample identification number, the volume of sample analyzed and the detector attenuation. Also, a sequential number should be assigned for each run made during a day's time. This can be useful for tracing carryover contamination problems in that it makes it easy to determine the analysis order even after the chromatograms have been separated. Integrator reports should also be labeled with the above information.

7.2 Storage

All data is to be stored in a neat, orderly fashion in one central location for easy future reference. All records should be maintained and stored on a daily basis to prevent a back-log of work and to facilitate in the early diagnosis of developing problems.

7.2.1 Prepare the chromatograms for storage.

7.2.1.1 Remove the chromatograms from the recorders.

7.2.1.2 Tear into appropriate lengths to fit the spectra binder notebook.

7.2.1.3 Reinforce the chromatogram ends with cellophane tape and punch holes to fit the binder.

NOTE: Corresponding FID and HECD chromatograms for each sample should be taped and stored together in the same binder.

7.2.2 Prepare the integrator reports for storage.

7.2.2.1 Underline and label identifiable peaks on the integrator reports.

7.2.2.2 Separate the reports.

7.2.2.3 Tape the corresponding FID and HECD reports for each sample on notebook paper.

7.2.2.4 Place the paper in a loose leaf binder with the FID and HECD reports on facing pages.

CAUTION

If the integrator reports are printed on thermal paper, do not place the cellophane tape over any important information as the tape will cause the printing to fade in a very short period of time. If this happens the information may become illegible.

7.2.3 Store the charts and tables.

7.2.3.1 Place the precision and accuracy quality control charts and the daily standard table in a loose leaf binder under appropriately titled sections.

7.2.3.2 Tape the calibration table in the sample log notebook.

7.3 Interpretation

Interpretation of data can be separated into two categories. One is the actual quantitation of sample data. This requires manipulation of the calibration curve data to determine compounds and amounts present. This is covered in more detail in the Standard Operating Procedure Quantitation of Water Samples. The second category of interpretations are qualitative in nature. These are primarily for quality control measures. Interpretations of this type are used to determine if the system is operating properly and if samples can be analyzed with the confidence of obtaining correct results. Purge blanks should have low backgrounds and the daily standards should give consistent area counts. The precision and accuracy charts should fall in the "in" control region. If any of the things mentioned above are askew, the possibility of system problems or operator error must be investigated.

7.3.1 Inspect the purge blank data daily.

7.3.2 Compare the daily standards.

7.3.3 Fill out the accuracy and precision charts.

NOTE: See Sections 8.1 and 8.2 for an explanation of these charts and how to fill them out.

8.0 QUALITY CONTROL

Steps must be taken to ensure the validity of all samples analyzed. This is accomplished by a rigorous quality control (QC) program. Quality control charts for accuracy and precision must be maintained. Also, chain-of-custody (COC) sheets must be filled out for each sample, blank and control analyzed. EPA quality control and performance evaluation samples are prepared and blindly analyzed interspersed with real water samples. A sample log must be kept indicating date, samples run, operators, and any problems encountered. This sample log is maintained in a brown RTI laboratory notebook.

8.1 Precision Chart

The precision chart (see Figure 5) is filled out daily using data acquired from a standard mixture. The daily response obtained with standard mixture B (Figure 6) is used for this purpose. This chart is a measure of the day-to-day reproducibility and is a guide for determining if the system is "in" or "out" of control. Samples may be analyzed if the value for   is less than 1.0. If the value exceeds 1.0 for several of the compounds, samples should not be analyzed.

8.1.1 Obtain one precision chart for each target compound.

8.1.2 Fill in the appropriate information at the top of the sheet.

8.1.3 Record the date and area count from the integrator report for the compound corresponding to the sheet.

NOTE: Area 1 corresponds to the area counts from the standard run the previous day and Area 2 corresponds to the current day's area count.

8.1.4 Perform the calculations indicated for each blank.

NOTE: RSD is the relative standard deviation. It is obtained from historical data for each compound.

8.1.5 Plot the number obtained for   on the graph.

8.1.6 If the point is below 0.77, write "in" in the space for condition.

8.1.7 If the point is between 0.77 and 1.0, write a "W" for warning in the space for condition.

8.1.8 If the point is above 1.0 write "out" in the space for condition.

CAUTION

If the condition is "out", the system is out-of-control. This means the reproducibility of the standard for that compound is poor compared to the previous day's run. If several compounds exhibit this condition, samples should not be analyzed until the problem is solved. Possible sources might be a bad injection, a bad standard, or other problems with the GC/purge and trap system.

QUALITY CONTROL CHART-PRECISION

TEAM WATER ANALYSIS

Target Compound: ______________________ **Gas Chromatograph:** ______________________

Relative Standard Deviation (RSD): ______________________ **Detector:** ______________________

DATE										
AREA(1)										
AREA(2)										
$\|1-2\|$										
SDU = (1)RSD										
R_c = 3.7(SDU)										
$\frac{\|1-2\|}{Rc}$										
Condition										

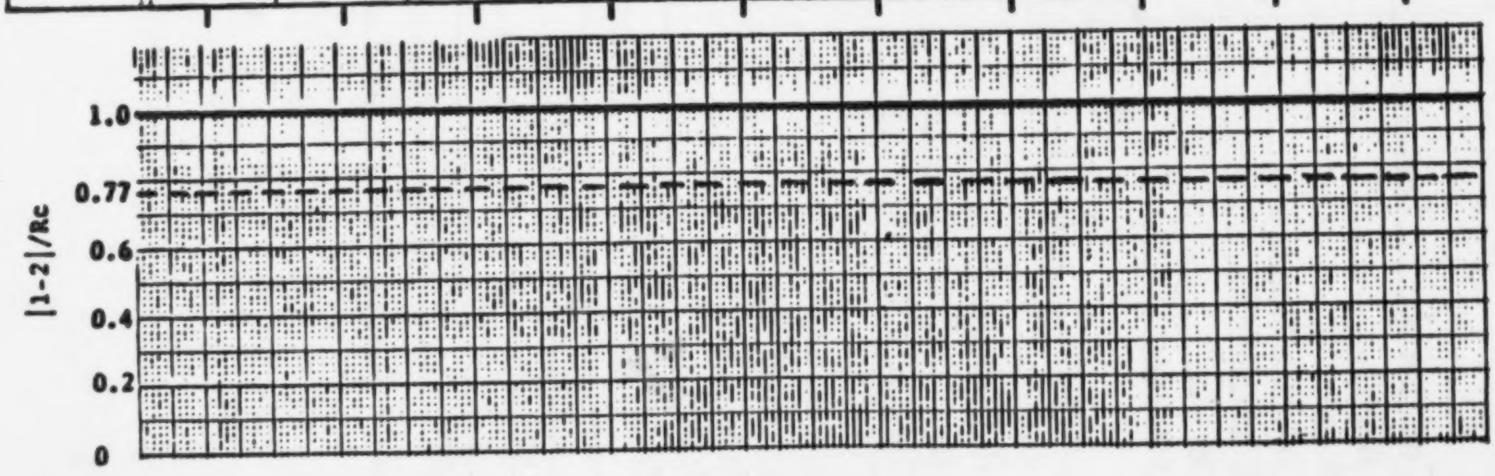

Figure 5. Quality control chart - Precision form.

TEAM DAILY RESPONSE TO STD ©

Compound Date:											
Aromatic					(integrator response x 10^4)						
Benzene											
Toluene											
Ethylbenzene											
Styrene											
p-Xylene											
Halocarbons											
VDC											
DCE											
MC											
CCl_4											
TCl											
[illegible]											
[illegible], $CHBr_3$											
PERC											
ClBen											
M-Cl_2Ben											
$CHCl_3$											
BDM											

Figure 6. TEAM daily response to standard mixture_C.

8.2 Accuracy Chart

The accuracy chart (see Figure 7) provides an indication of the reliability of the compound quantities based on the results from the daily standards. Quantitation is acceptable if the value for is between -2 and +2, warning if the value is between +2 and +3 or -2 and -3, and unacceptable if greater than +3 or less than -3. If the value indicates the accuracy to be unacceptable for several compounds, samples should not be analyzed until the problem is solved.

8.2.1 Obtain one accuracy chart for each target compound.

8.2.2 Fill in the appropriate information at the top of the sheet.

NOTE: The values for CAR (calibration area response) are obtained by calculating the area response at the concentration of the daily standard using the most recent calibration curve. The value for SDU is transferred from the precision charts.

8.2.3 Record the date and area count from the integrator report.

8.2.4 Perform the indicated calculations and fill in the spaces provided.

8.2.5 Plot the number obtained for on the graph.

8.2.6 If the point is between -2 and +2 write "IN" in the space for the condition.

8.2.7 If the point is between -3 and -2 or +2 and +3 write a "W" for warning in the space for condition.

8.2.8 If the point is greater than +3 or less than -3, write "OUT" in the space for condition.

CAUTION

If the condition is "OUT", the reliability of the quantitation is questionable. If this occurs for several compounds the problem should be investigated and solved before beginning sample analysis.

8.3 Chain-of-Custody Sheets

Chain-of-custody (COC) sheets (see Figure 8) must be filled out for each sample analyzed. Fill out each corresponding sheet as soon as the sample is analyzed.

QUALITY CONTROL CHART-ACCURACY

TEAM WATER ANALYSIS

Target Compound: ____________________ **Gas Chromatograph:** ____________________

Date Calibration Run: ____________________ **Detector:** ____________________

Calibration Area Response (CAR): ____________________ **Concentration:** ____________________

% Relative Standard Deviation: ____________________ **Standard Deviation Unit (SDU):** ____________________

DATE										
AREA(A)										
A-CAR										
$\frac{\text{A-CAR}}{\text{SDU}}$										
Condition										

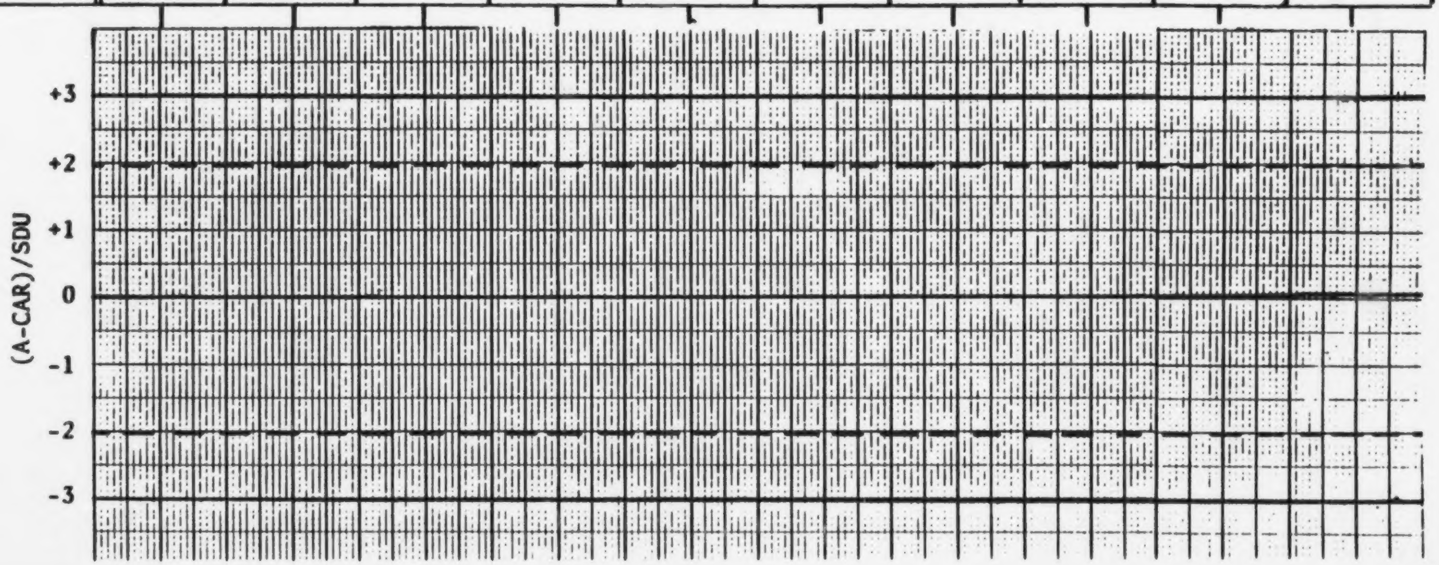

Figure 7. Quality control accuracy form.

CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute, Analytical Sciences Division
Chemistry and Life Sciences Group, Research Triangle Park, NC 27709

SAMPLE CODE	TENAX BATCH:	VOLUME COLLECTED:
SAMPLE TYPE:	TEMPERATURE (°F)	VOLUME ANALYZED:

RELINQUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED

Figure 8. Chain-of-custody record form.

8.3.1 Locate the sheet that goes with the sample being analyzed.

NOTE: For the water samples, it is generally 25.0 mL; however, if 25.0 mL was not analyzed for some reason, that reason must be explained on the COC sheets and the volume analyzed written in the "volume analyzed" space.

8.3.3 Initial the Rec'd and Relinquished spaces.

8.3.4 Write the date analyzed in the space provided.

8.3.5 Write "Analyzed by P+T" in the space labeled "Operation Performed."

8.3.6 Make notations of any abnormalities at the bottom of the sheet.

8.4 Sample Log

A sample log is to be kept with entries indicating the samples run, the daily standard run, the operators names, problems encountered and the date. This is to be kept in a brown RTI Laboratory notebook.

8.5 Performance Evaluation Samples and Quality Control Samples

Performance evaluation and quality control samples are prepared from concentrates available from EPA. The concentrated spiking solutions contain halogenated and aromatic compounds and are sealed in glass ampules. Performance evaluation concentrates 1 and 2 contain eleven halogenated organic compounds and concentrates 3 and 4 contain six aromatic compounds. The quality control ampules contain nine halogenated organic compounds. Organic-free water is spiked using the above solutions and placed in water bottles identical to those used for water samples. The synthetic samples are labeled as if they were duplicate water samples. The preparation of these synthetic samples should be performed by someone other than the analysts to insure these being "Blind" samples. These samples are interspersed with the real samples and are analyzed unknowingly by the analysts. The results of these samples are reviewed by the QA Officer.

8.5.1 Acquire ampules and stabilize at 20°C.

8.5.2 Add 100 mL of organic free water to a 100 mL volumetric flask.

8.5.3 Open the ampule and transfer 5 µL of the contents using a 10 µL syringe.

NOTE: Inject the solution just under the surface of the water.

8.5.4 Invert the flask 3 times to mix.

8.5.5 Transfer the spiked water to 60 mL amber water bottles.

8.5.6 Label and fill out COC sheets to simulate the real water samples.

8.5.7 Give a list of the identification codes used on these samples to the QA Officer.

8.5.8 Store the synthetic samples with the real samples.

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Standard Operating Procedure for the Analysis of Volatile Organics Collected on Tenax Using the Finnigan 4021 GC/MS/DS

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Standard Operating Procedure for the Analysis of Volatile Organics Collected on Tenax Using the Finnigan 4021 GC/MS/DS

RTI/ACS-SOP-533-01

1.0 SCOPE AND APPLICATION

This standard operating procedure is applicable to the qualitative and quantitative analysis of volatile organic compounds collected on Tenax GC®. The method is not applicable as herein described to the analysis of permanent gases present in the atmosphere. In general, recovery from Tenax for compounds less volatile than ten-twelve carbon hydrocarbons is less than quantitative, so the method is not applicable to the quantitative analysis of the less volatile compounds, although many compounds of low volatility can be observed by thermal desorption from Tenax and analyzed qualitatively. Thermal desorption of Tenax followed by cryofocussing of the organic vapors, with subsequent capillary gas chromatography/mass spectrometry/data system analysis has been applied to adsorbed volatile organic compounds collected from exterior and interior air, personal air, air collected in the workplace, breath, and volatile organics transferred to Tenax from other adsorptive media. The method is limited by adsorption of water on the Tenax: if too much water is adsorbed, the cryofocussing trap will freeze and quantitative analysis will be impossible. The basic method is adaptable to any gas chromatograph/mass spectrometer/computer system upon construction of a suitable thermal desorption unit. A certain amount of flexibility in the analytical method from instrument to instrument is tolerable in order to optimize the operating parameters of any given instrument. Data handling procedures may also follow a wide range (from completely computerized to entirely manual) and still produce data within the criteria for acceptability. The following Standard Operating Procedure is related to optimal operating procedures for a particular instrument.

2.0 SUMMARY OF THE PROCEDURE

Tenax GC® is a porous p-phenylene oxide polymer which preferentially adsorbs volatile organic compounds. The affinity of this polymer for water is low, and thus it is possible to sample many liters of air (often 20-30 or more) without adsorbing so much water that application of analytical procedures becomes prohibitive. The affinity of this polymer for organic vapors makes its cleaning and preparation for field sampling crucial to the success of the ultimate analysis. Once the sample cartridge has been exposed in the field, extremely careful handling is required in order to avoid contamination. The sample cartridge, shipped back from the field, is loaded with a standard (or standards) to permit quantitative analysis. The cartridge is then submitted for analysis by capillary gas chromatograph/mass spectrometer/data system. The organic vapors are removed from the Tenax by heating the sample cartridge under a flow of helium. The desorbed vapors are collected in a trap which is cooled to liquid nitrogen temperatures. This step is essential to focus the organic compounds and allow their application to the head of the capillary column in a discrete band. A cartridge heater raises the temperature of the trap from liquid nitrogen temperatures (-198°C) to 250°C in approximately 2.5 minutes, vaporizing all of the entrapped organics very quickly. The vaporized organics are transferred to the head of the capillary column which is at or near room temperature, the scan of the mass spectrometer is initiated, and the analytical procedure is begun. The column is programmed to a temperature to allow the elution of all of the organic compounds while the mass spectrometer is scanning. Data are recorded by the computer for subsequent processing. Quantitation is performed by the method of Relative Response Factors, where the proportionate system responses for analyte and standard are determined prior to the analysis of the sample and this relative system response is used to determine the quantity of compound present on the sample cartridge. The quantitative analysis is performed by a combination of manual and computerized procedures: the computer is instructed to seek characteristic ions in a previously determined retention window. At this point the operator intervenes to determine if the compound of interest has been located correctly. If the compound identification is correct, the computer then performs the quantitative calculation using the method of Relative Response Factors. Data are reported as ng/cartridge, and can be subsequently converted to whatever units are desired.

Quality control procedures are followed in order to determine that the column is performing within acceptable limits, the mass spectrometer is tuned correctly and performing acceptably and chromatography criteria are being met. A chromatogram (actually reconstructed ion chromatogram) is obtained for each analysis and entered into the laboratory project notebook. The quantitation report from the computer is also entered into the project notebook. Standard Chain-of-Custody procedures are followed for every sample analyzed. The deliverables include a copy of the computer-generated quantitation report and the Chain-of-Custody form. Also, an entry is made into the appropriate instrument log for every sample analyzed.

3.0 PREPARATION OF GOLD-PLATED NICKEL TUBING

Gold-plated nickel tubing is required for an inert transfer line between the desorption unit and the capillary column. This tubing must be prepared, as it is not commercially available.

3.1 Materials and Equipment

- 3.1.1 1/16" o.d. nickel tubing (15") (Supelco)
- 3.1.2 Aspirator (Corning)
- 3.1.3 Large beaker (Corning)
- 3.1.4 Heater (Fisher)
- 3.1.5 Teflon tubing (Penntube)
- 3.1.6 Atomex (AuCN) (Englehard)
- 3.1.7 Distilled water (Research Triangle Institute)
- 3.1.8 Acetone (Fisher)
- 3.1.9 20 mL 1N HCl (Fisher)
- 3.1.10 Thermometer (Supelco)
- 3.1.11 Solution of 10% OV-17 in acetone (Supelco)

3.2 Procedure

3.2.1 Fill large beaker with water and submerge the coiled nickel tubing (see Figure 1).

3.2.2 Using the aspirator, pull 20-40 mL acetone through the tubing.

NOTE: This rinse with acetone will remove trace organic compounds from the tubing.

3.2.3 Heat water bath to 70°C.

3.2.4 Pull 20 mL 1N HCl through the nickel tubing.

3.2.5 Rinse the acid from the tubing by pulling through 50 mL distilled water.

3.2.6 Draw 20 mL of Atomex through the tubing at a rate of 1 mL/min.

NOTE: This amount of Atomex is only an estimate; more or less may be required to complete the plating process. The amount of plating can be checked by inspecting the end of the tubing immersed in the Atomex which was last reached by the Atomex.

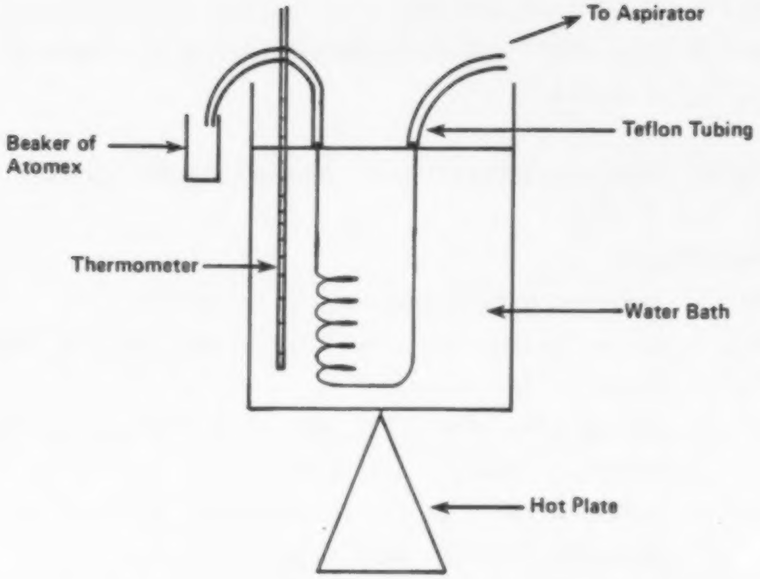

To Aspirator
Beaker of Atomex
Teflon Tubing
Thermometer
Water Bath
Hot Plate

Figure 1. Diagram of gold plating nickel tubing.

3.2.7 Rinse the tubing with distilled water, HCl, and distilled water again.

WARNING

Be extremely careful not to allow any mixing of Atomex (AuCN) with any of the HCl rinses in the aspirator flask. If Atomex and HCl are allowed to mix, toxic HCN is formed.

3.2.8 Rinse with acetone.

3.2.9 Pull OV-17 solution through tubing at a rate of approximately 1 mL/min.

4.0 INSPECTION AND PREPARATION OF THERMAL DESORPTION UNIT

4.1 Materials

4.1.1 Six-port high temperature valve (Valco)

4.1.2 25 mm Teflon cap, 5 mm thick (fabricated at Research Triangle Institute)

4.1.3 Nickel trap with 1/16" o.d. nickel tubing (fabricated at Research Triangle Institute)

4.1.4 Aluminum block to accept cartridge (fabricated at the Research Triangle Institute)

4.1.5 Block heaters (2) (Hotwatt)

4.1.6 Thermocouples (6) (fabricated at the Research Triangle Institute)

4.1.7 1/16" male nuts (6) (Valco)

4.1.8 1/16" ferrules (6) (Valco)

4.1.9 Desorption chamber and cap handle (fabricated at the Research Triangle Institute)

4.1.10 Aluminum foil (grocery store)

4.1.11 Cotter pin (hardware store)

4.1.12 Insulation

4.1.13 Heater for transfer line (fabricated at the Research Triangle Institute)

4.1.14 Valve stem and handle (Valco)

4.1.15 Front and back cover plates (fabricated at the Research Triangle Institute)

- 4.1.16 Screws (hardware store)
- 4.1.17 Melting point capillary tubes (Supelco)
- 4.1.18 1/16" o.d. stainless steel tubing (8") (Supelco)
- 4.1.19 1/16" o.d. gold-plated nickel tubing (15") (tubing from Supelco, gold-plated at the Research Triangle Institute)
- 4.1.20 Allen wrench
- 4.1.21 Flat-bladed screwdriver (Sears)
- 4.1.22 7/16" open-end wrench (Sears)
- 4.1.23 Temperature controller for thermal desorption unit (fabricated at the Research Triangle Institute)
- 4.1.24 Metal mounting bracket for thermal desorption unit, to mount unit to the gas chromatograph (fabricated at the Research Triangle Institute)
- 4.1.25 1/8" copper tubing (several feet) (Dillon Supply)
- 4.1.26 Fine metering valve (Hoke)
- 4.1.27 Toggle valve (Raleigh Valve and Fitting)
- 4.1.28 Helium (2 tanks) (National Welders)
- 4.1.29 Helium regulators (2) (Matheson)
- 4.1.30 T-bar (Swagelok)
- 4.1.31 1/4"-1/8" female pipe fitting (2) (Swagelok)
- 4.1.32 Bubble flow meter (Supelco)
- 4.1.33 Metal column tags (Supelco)
- 4.1.34 Snoop (Nupro)

4.2 Disassembly of the Thermal Desorption Unit to Permit Inspection

4.2.1 Remove the back cover plate by extracting four screws with a flat-bladed screwdriver.

NOTE: With the cover plate removed, the inner fiberglass insulation can be removed. Use gloves when removing the insulation. Particles of fiberglass on the skin will be irritating.

4.2.2 Extract the insulation, setting it carefully aside for later reinsertion, to make the back half of the aluminum block visible.

4.2.3 Dismount the desorption unit from above the oven of the gas chromatograph by removing two screws connected to its base with a flat-bladed screwdriver.

4.2.4 Lay the dismounted unit face down on a table, being very careful not to displace any thermocouple or heater wires.

4.2.5 Using a 5/32" Allen wrench, remove the four bolts from the aluminum block to allow the six-port high temperature valve and all of its associated connections (see Figure 2) to be visible.

4.2.6 Now that the unit has been disassembled to this point, initiate the inspection.

4.3 Inspection of the Thermal Desorption Unit

4.3.1 Verify that all fittings which connect to the six-port valve are tight, using a wrench to tighten them if necessary.

4.3.2 Verify that a heater and two thermocouples are wrapped securely with aluminum foil around the gold-plated transfer line.

4.3.3 Check that the thermocouples are encased in a short length of glass capillary tubing to prevent shorting out against metal surfaces and loss of a temperature reading.

4.3.4 Verify that two thermocouples encased in glass melting point capillaries are located on the side of the front piece of the aluminum block.

NOTE: The unit will not give temperature readouts which are crucial to the maintenance of proper operating temperatures if the thermocouples are not securely encased in glass or if the glass is cracked. These glass tubes must cover the length of exposed wire of the thermocouple.

4.3.5 Check that the nickel lines connected to the valve and extending to the trap are covered with aluminum foil until the lines start to coil at the trap.

4.3.6 Verify that a heater is located at the top of the trap.

4.3.7 Check to be certain that the "trap in" line actually goes down to the bottom of the trap, and that the "trap out" line coils back up.

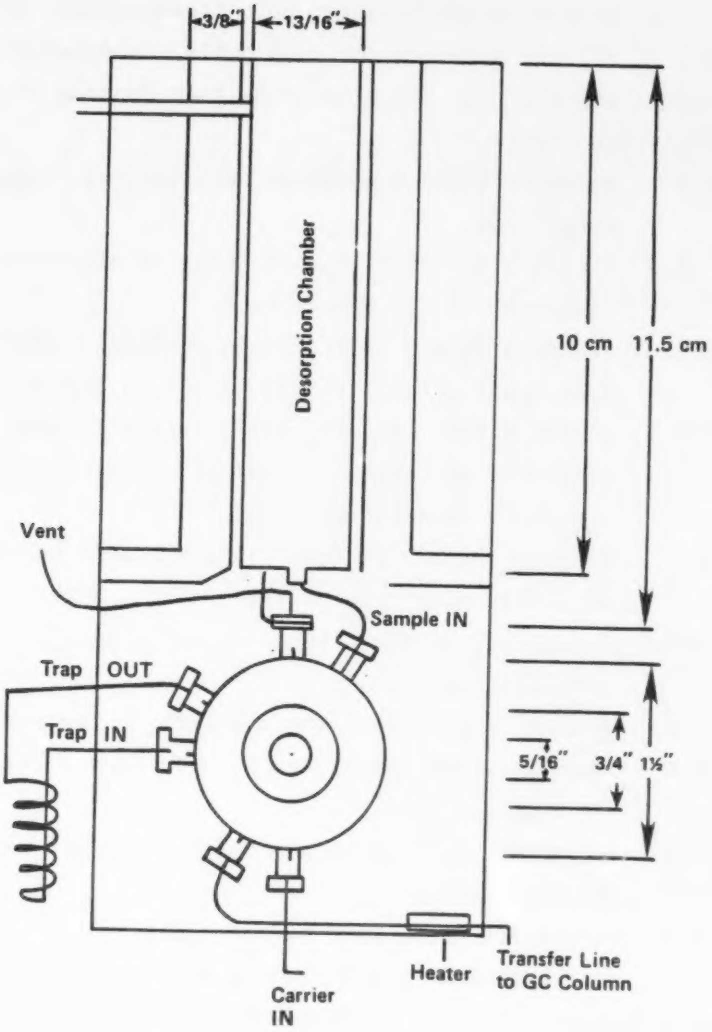

3/8"
13/16"
Desorption Chamber
10 cm
11.5 cm
Vent
Sample IN
Trap OUT
Trap IN
5/16"
3/4"
1½"
Heater
Transfer Line to GC Column
Carrier IN

Figure 2. 6-Port Valco high temperature valve and connections.

4.3.8 After inspection of all the areas indicated above, reinstall the desorption unit above the gas chromatograph oven with the transfer line going through a hole in the gas chromatograph injector port block closest to the ion source.

4.3.9 At this point do not reassemble the desorption unit.

4.4 Installation of Gas Flows to Allow Leak-Testing of the Thermal Desorption Unit

4.4.1 To inspect the desorption unit further, connect the helium purge flow.

4.4.2 Attach a 1/4"-1/8" female Swagelok pipe fitting to the regulator of the helium tank.

4.4.3 Attach a length of 1/8" copper tubing sufficient to reach from the regulator to the mass flow controller.

4.4.4 Insert a fine metering valve in this copper line near the regulator and insert an on/off toggle valve in the copper line near the desorption unit.

4.4.5 Connect the end of the line subsequent to the toggle valve to a mass flow controller.

4.4.6 Using 1/8" copper tubing, connect the flow controller to the desorption unit.

4.4.7 Set the pressure of the regulator at approximately 40 psi.

4.4.8 Open the tank valve and the regulator valve to allow helium to fill the line.

4.4.9 Regulate the flow of gas to 15-20 mL/min using the fine metering valve.

4.4.10 Verify the flow rate by attaching a bubble flow meter to the stainless steel "helium out" line of the desorption unit.

4.4.11 Label this gas line with a metal tag reading "Helium purge line."

NOTE: In order to check that the desorption unit is leak-tight in both "Desorb" and "Inject" positions, it is necessary also to connect the carrier gas.

4.4.12 Connect a 1/4"-1/8" female Swagelok pipe fitting to a helium tank.

4.4.13 Set the regulator to a pressure reading of 40 psi.

4.4.14 Connect a length of 1/8" copper tubing to this fitting.

4.4.15 Label this line "Carrier Gas" with a metal tag.

4.4.16 On the Finnigan 4021, connect the carrier gas line at the "Helium in" position of the CI controller panel (see Figure 3).

4.4.17 Connect another 1/8" copper line to a T-bar off the "Helium In" line.

4.4.18 Connect this branched line to a Matheson Helium Mass Flow Controller Model 8240 at the threaded fitting labeled IN.

4.4.19 Attach an 1/8" copper line to the OUT position of the flow controller which leads to the "Helium In" line of the six-port valve.

4.4.20 Open the valve of the helium tank regulator.

4.4.21 Turn on the POWER switch of the flow controller.

4.4.22 Set the toggle switch to the CONTROL setting.

NOTE: Once the POWER of the flow controller is ON, it is essential to keep a flow of helium through the controller. Leaving the flow controller ON without a flow of helium will result in burning out the regulator heads of the flow controller.

4.4.23 Adjust the black grooved knob centered on the front panel of the flow controller until the digital readout is 1.5 mL/min.

4.4.24 Verify this reading with a bubble flow meter to ensure that the flow controller is working properly.

4.5 Leak Testing of the Thermal Desorption Unit

4.5.1 Check the six-port valve for leaks in the connections after the gas flows have been connected.

4.5.2 Check the valve in both the DESORB and INJECT positions.

4.5.3 Cap off the transfer line and vent line so that no gas flow can exit from the valve.

4.5.4 Turn the handle of the valve to the DESORB mode (up).

4.5.5 Turn up the flow controller to approximately 5.0 mL/min.

NOTE: The reading on the flow controller will remain at 5.0 mL/min for approximately 1 to 2 minutes and should then drop quickly. If the digital readout of the flow controller does not drop to zero or a negative number, either the body of the valve, the connections to the valve, the Teflon cap of the desorption unit (or some combination of the three) is leaking.

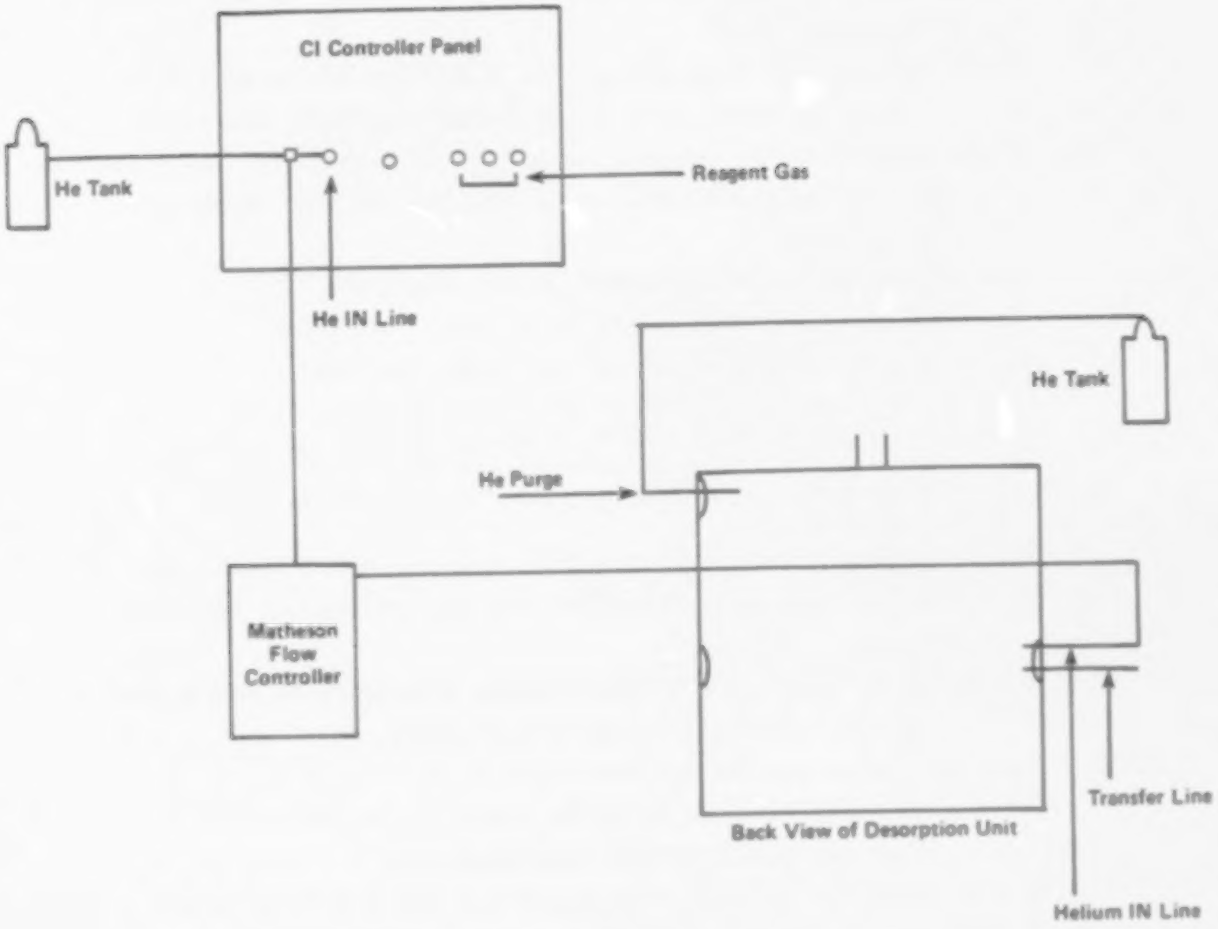

CI Controller Panel
He Tank
Reagent Gas
He IN Line
He Tank
He Purge
Matheson Flow Controller
Transfer Line
Back View of Desorption Unit
Helium IN Line

Figure 3. Carrier and purge gas flow to the thermal desorption unit.

4.5.6 Examine the final reading of the flow controller to determine the severity of the leak.

4.5.7 Inspect all connections going into the desorption unit and six-port valve for leaks by putting Snoop on the connections.

NOTE: Bubbles will appear immediately at a leaking connection.

4.5.8 Allow the Snoop to remain on the fittings under observation for several minutes because a slow leak may be present.

4.5.9 Tighten the appropriate fitting(s) if a leak is found.

4.5.10.Recheck the unit by repressurizing and following the above leak-checking sequence.

NOTE: If tightening a fitting does not produce a leak-tight unit, the nut and ferrule may not be seated properly. Loosen the nut and remove the line from the six-port valve. Cut the line behind the nut so that a new ferrule can be installed and seated. If this cutting will result in a line which will be too short to use after cutting, it will be necessary to use a new length of nickel tubing.

NOTE: Do not use a tubing cutter on nickel tubing. The tubing cutter will deform or even partially seal the severed line in the process of cutting, leaving an opening with a greatly reduced diameter which will act as a restrictor to gas flow. Cut the tubing by scoring on one side with a file and snapping off the desired length of tubing.

NOTE: Inspect the nut cut from the line carefully in order to be certain that the threads are in sufficiently good condition to allow reuse. If this nut cannot be reused, obtain a new nut in order to obtain an effective seal.

4.6 Seating Ferrules

4.6.1 Since the use of a new valve to seat a ferrule could result in a nut breaking off inside the valve and rendering it unfit for use, seat a ferrule in an old valve or other appropriate fitting.

4.6.2 Place a 1/16" Valco nut and ferrule on the metal line.

4.6.3 Insert the line into a threaded opening in the old valve or fitting.

4.6.4 Tighten the nut with a 1/4" wrench.

4.6.5 Verify that the ferrule is seated by checking whether the ferrule moves on the line: a properly seated ferrule will not move.

4.6.6 When the ferrule has been seated correctly, reinstall the line on the six-port valve and repeat the leak-checking procedure.

4.7 Leak-Testing the Teflon Cap

4.7.1 Inspect the Teflon cap of the thermal desorption unit for leaks with the valve set in the DESORB mode.

4.7.2 Apply Snoop on top of the cap as well as around it.

4.7.3 If a leak is not observed instantaneously, leave the Snoop on for several minutes to check for a slow leak.

4.7.4 If a leak is found, replace the Teflon cap with a new one and repeat the leak-checking procedure.

4.8 Replacing the Teflon Cap of the Thermal Desorption Unit

4.8.1 Remove the two screws attached to the thermal desorption chamber which hold the cap assembly on the desorption unit with a flat-bladed screwdriver.

NOTE: The entire cap handle can now be removed.

4.8.2 Inspect underneath the Teflon cap to verify the presence of a brass disk with a spring soldered to a screw.

4.8.3 Loosen the screw to allow the cap and washer to be removed.

4.8.4 Center the washer on the new Teflon cap (25 mm o.d. x 5 mm thick).

4.8.5 Screw the spring soldered to a screw back onto the unit.

4.8.6 Put the handle assembly back on top of the thermal desorption unit.

4.8.7 Tighten the two side screws with a flat-bladed screwdriver.

4.8.8 Center the washer and cap on the top of the thermal desorption unit.

4.8.9 Push the handle down to allow the cap and desorption chamber to be flush with each other.

4.8.10 Repeat the leak-checking procedure.

4.8.11 If the cap does not now seal tightly, install another small washer above the washer already on top of the cap.

4.8.12 Repeat the leak-checking procedure.

4.8.13 If the cap still does not seal tightly, repeat the procedure to install a new cap.

4.8.14 Repeat the leak-testing procedure.

4.9 Reassembly of the Desorption Unit

4.9.1 When all connections to the valve and the Teflon cap of the thermal desorption unit have been inspected and no leaks have been found to be present, reassemble the thermal desorption unit.

4.9.2 Screw the back half of the aluminum block into place using a 5/32" Allen wrench on the four bolts.

4.9.3 Recheck the thermocouples for the block to verify that they are securely in place.

4.9.4 Check again to be sure that the thermocouples are insulated by glass capillary tubes on their metal ends.

4.9.5 Insert the insulation around the aluminum block (with gloves on) being careful not to dislodge any thermocouple or heater wires.

4.9.6 Connect the back panel to the thermal desorption unit.

5.0 ADJUSTMENT OF GAS FLOWS

The flows for the Helium purge and Helium carrier gas have already been adjusted to 15.0 mL/min and 1.0 mL/min, respectively, in order to test the thermal desorption unit for leaks. The other gas flow needed to operate the thermal desorption unit is a nitrogen flow into the nickel trap.

5.1 Materials and Equipment

The materials and equipment needed to set up and adjust the gas flows for operation of the thermal desorption unit are listed below.

5.1.1 Nitrogen (1 tank) (National Welders)
5.1.2 Nitrogen regulator (Matheson)
5.1.3 1/4"-1/8" female pipe fitting (Swagelok)
5.1.4 1/8" copper tubing (several feet) (Dillon Supply)
5.1.5 1/8" nuts and ferrules (4) (Swagelok)
5.1.6 1/4" copper tubing (approximately 1 foot) (Dillon Supply)
5.1.7 1/4" nuts and ferrules (2) (Swagelok)
5.1.8 1/4" to 1/8" reducing unions (2) (Swagelok)
5.1.9 ON/OFF toggle valve (Raleigh Valve and Fitting)
5.1.10 Dewar flask (Fisher)

5.2 Assembling Gas Lines and Setting Flows

5.2.1 Attach a 1/4" to 1/8" female pipe fitting to the regulator of the nitrogen tank.

5.2.2 Set the regulator of the nitrogen tank to read a pressure of approximately 60-70 psi.

5.2.3 Attach an 1/8" copper line to the pipe fitting with 1/8" nuts and ferrules.

5.2.4 Put a toggle valve into the copper line within easy reach of the thermal desorption unit.

5.2.5 Attach the 1/8" copper line to the 1/4" copper line.

5.2.6 Allow two or three coils of the 1/4" copper line to rest at the bottom of the Dewar flask.

5.2.7 Attach the 1/4" copper line to the 1/8" copper line.

5.2.8 Terminate the copper tubing at the nickel trap (see Figure 4).

6.0 ADJUSTMENT OF DESORPTION UNIT TEMPERATURES

All temperatures within the thermal desorption unit must be monitored very closely during their initial heat-up period to verify that the potentiometers on the control unit are adjusted correctly and that the thermocouples within the desorption unit are functioning properly.

6.1 Temperature of the Desorption Unit Aluminum Block

6.1.1 After the flow of helium to the six-port valve is established, heat the aluminum block to 270°C.

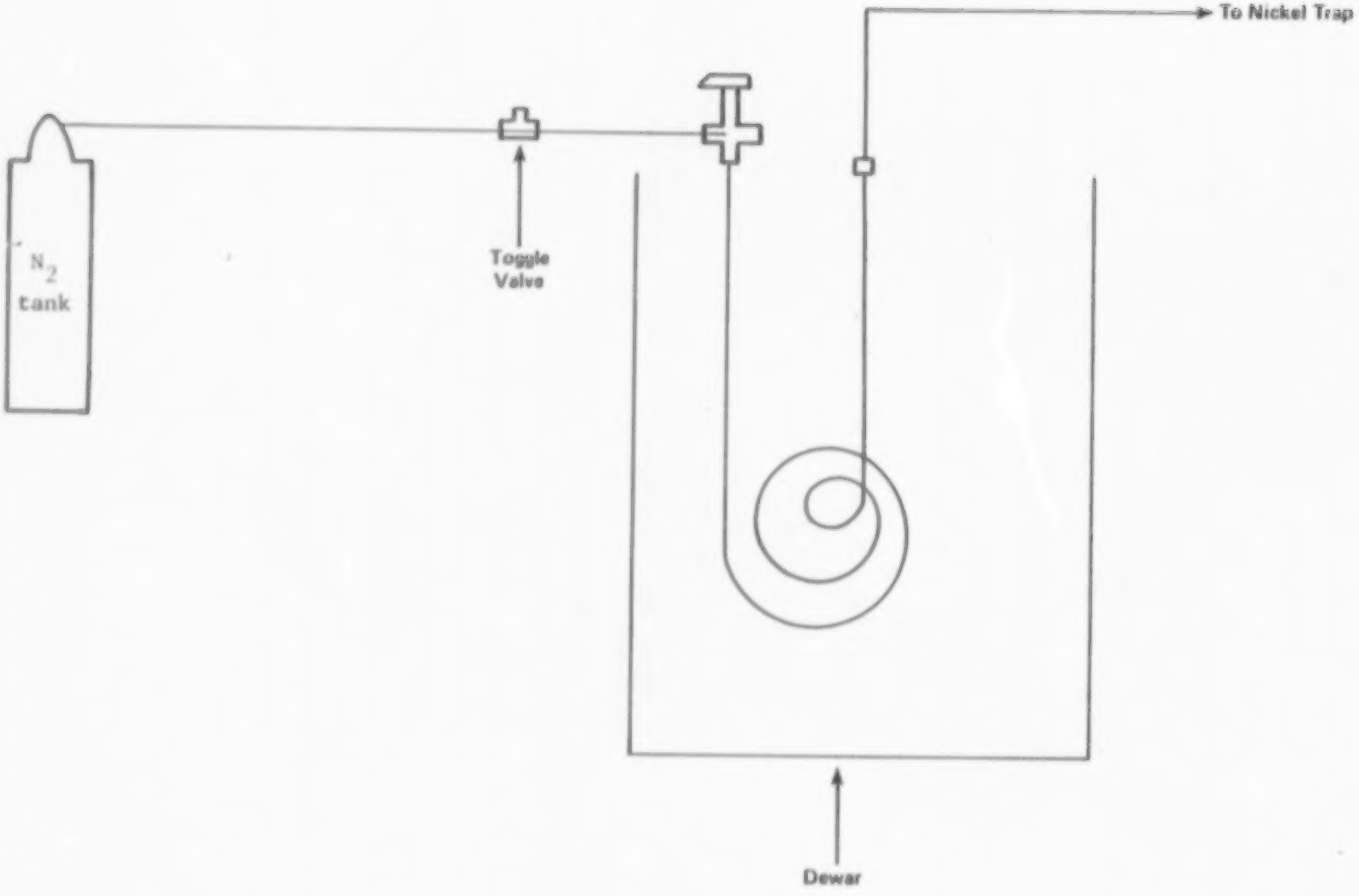

N_2 tank
Toggle Valve
To Nickel Trap
Dewar

Figure 4. Nitrogen gas flow.

6.1.2 To heat the aluminum block, turn on the toggle switch labeled "Desorption Unit Block" located on a metal panel beneath the gas chromatograph.

6.1.3 Turn the metal knob beside the digital readout panel to the first position in order to monitor the temperature of the block.

6.1.4 Verify that the red light adjacent to the toggle switch is lit when the toggle switch is on.

6.1.5 Turn the black potentiometer clockwise to increase the temperature and counterclockwise to decrease the temperature.

NOTE: An orange light above the black potentiometer will be lit when the aluminum block is heating.

6.1.6 While the block is heating, rotate the six-port valve frequently from the DESORB mode to the INJECT mode and back again.

NOTE: The valve should rotate smoothly at 270°C.

NOTE: If the valve is not rotated during heating, it will most probably freeze and will not be able to be rotated at all. If the valve freezes, cool the aluminum block to room temperature and try gently to rotate the valve. If the valve remains frozen, disassemble the thermal desorption unit completely, remove the frozen valve, install a new valve, and repeat the leak-checking and reassembly procedures.

6.2 Adjustment of the Temperature of the Gold-Plated Transfer Line

6.2.1 While the aluminum block is heating, heat the gold-plated transfer line from the six-port valve to 240°C.

NOTE: The controls for this transfer line are located and labeled on the same panel as the controls for the aluminum block and the temperature of the gold-plated transfer line can be monitored by turning the metal knob to the third position.

6.3 Temperature Regulation of the Nickel Trap

6.3.1 Monitor the temperature of the nickel trap in the second position of the metal knob.

6.3.2 Set the potentiometer so that the heating is halted at 240°C.

6.3.3 Allow the trap to go through at least one heating and cooling cycle to verify that it is functioning properly.

NOTE: Often, even when the heating is manually turned off at 240°C, the nickel trap will overshoot to approximately 255°C.

7.0 INSTALLATION OF FUSED SILICA CAPILLARY COLUMNS

Capillary gas chromatography is used in the analysis of organic compounds desorbed from Tenax cartridges in order to attain the maximum sensitivity and resolution.

7.1 Materials and Equipment

7.1.1 50 or 60 m wide-bore fused silica capillary column, either thick film coating of phase or chemically bonded (American Scientific Products)
7.1.2 1/4" open-end wrenches (2) (Sears)
7.1.3 0.4 mm i.d. graphite ferrule (2) (Supelco)
7.1.4 0.4 mm i.d. graphite/Vespel ferrule (1) (Supelco)
7.1.5 1/16" stainless steel back ferrule (2) (Raleigh Valve and Fitting)
7.1.6 1/16" stainless steel front ferrule (1) (Raleigh Valve and Fitting)
7.1.7 Zero dead volume fitting (1) (Swagelok)
7.1.8 1/16" threaded male nut (2) (Swagelok)
7.1.9 Old 10 microliter syringe (Supelco)
7.1.10 3 cm o.d., 1.5 cm thick high temperature septum (Supelco)
7.1.11 1/16" stainless steel female nut (Swagelok)
7.1.12 5/16" open end wrench (Sears)

7.2 Procedure

7.2.1 Verify that a 1/16" Swagelok male nut with 1/16" back and front ferrule can be slipped over the gold-plated nickel transfer line from the six-port valve.

7.2.2 If the fittings cannot be slipped over the transfer line, carefully file the end of the line so the fittings can be slipped over it.

7.2.3 Attach the zero dead volume fitting, being certain to seat the ferrule properly.

7.2.4 Remove one loop of the fused silica capillary column from the closed side of the column cage.

7.2.5 Remove three loops from the open side.

7.2.6 Remove the cover from the transfer line oven.

7.2.7 Insert the length of column equivalent to three loops through a small opening at the rear of the oven of the gas chromatograph.

7.2.8 Pull the column end up into the transfer line oven.

7.2.9 Put the column cage on the metal column holding bar in the oven of the gas chromatograph.

7.2.10 In this order, carefully put on the front end of the column: 1/16" male threaded nut, 1/16" stainless steel back ferrule, 3 cm o.d. high temperature septum, and a 0.4 mm i.d. graphite ferrule.

NOTE: The order is critical to seal the column properly in the zero dead volume fitting (see Figure 5).

7.2.11 If the column cannot be pushed through the opening in the ferrule, bore out the graphite ferrule with an old 10 microliter syringe needle.

7.2.12 Once the graphite ferrule is on the end of the column, break off about 2-3 inches of column to ensure that no graphite ferrule particles have blocked the opening of the column.

7.2.13 Be sure that the break is clean with no rough edges.

7.2.14 Position and hold the column with one hand while sliding the nut up to the zero dead volume fitting.

7.2.15 Thread and tighten the nut while holding the column in position with the other hand, holding the column until the nut is finger-tight, at which time the column will stay in position without being held.

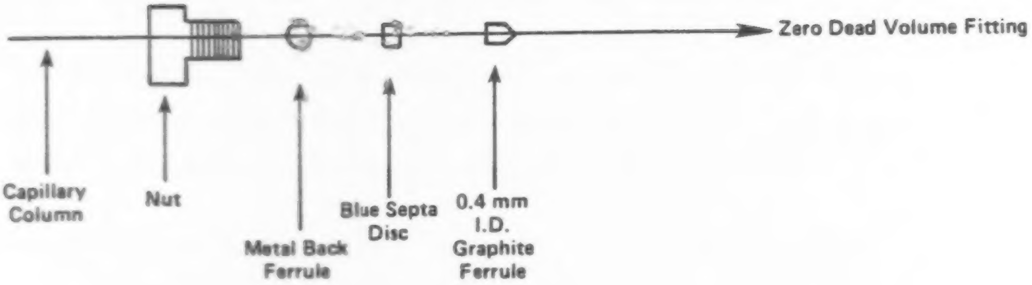

Zero Dead Volume Fitting
Capillary Column
Nut
Metal Back Ferrule
Blue Septa Disc
0.4 mm I.D. Graphite Ferrule

Figure 5. Front end of GC column.

7.2.16 Continue tightening the nut with two 1/4" open-end wrenches, one wrench holding the zero dead volume fitting and the other turning the nut.

7.2.17 Tighten the nut until it feels snug.

7.2.18 Before connecting the back end of the column, check the helium carrier gas flow to verify that helium is flowing through the column.

NOTE: If the column has not been conditioned, do not connect the back end of the column. Follow the column manufacturer's directions for conditioning the column, and connect the back end only after the column has been conditioned.

7.2.19 Check for gas flow through the capillary column by filling a small vial with water and putting the back end of the column into the water.

NOTE: If flow is present, bubbles will be observed.

7.2.20 If no bubbles are observed, verify that the helium tank valve is open, the flow controller is turned on and the flow controller is set at 1.0 mL/min.

7.2.21 If there is still no flow, check whether the column has broken off inside the zero dead volume fitting.

7.2.22 Once flow through the column has been established, put a 1/16" stainless steel female nut with 0.4 mm i.d. graphite/Vespel ferrule on the back end of the column.

7.2.23 Bore out the graphite/Vespel with the old syringe needle to allow the ferrule to fit over the end of the column.

7.2.24 Push the ferrule onto the end of the column approximately 30 cm.

7.2.25 Break off approximately 2 inches of the column to ensure that no particles of the ferrule have entered the column to block the flow of helium.

7.2.26 Holding the column with one hand, thread and finger-tighten female nut to the threaded male fitting located in the transfer line over on the side nearest the ion source.

7.2.27 Position the end of the column in the ion source relative to the filament by observing through the plexiglass plate on top of the ion source.

7.2.28 Two filament copper leads are attached to separate filament posts; push the column through the threaded fitting in the transfer line oven until the end of the column can be positioned between the two filament posts (see Figure 6).

7.2.29 Tighten the nut snugly with 5/16" open-end wrench.

8.0 STARTING THE VACUUM SYSTEM OF THE MASS SPECTROMETER

When all fittings are air-tight, start the vacuum system of the mass spectrometer to produce the high vacuum essential for operation. The mechanical and diffusion pumps of the mass spectrometer are assumed to be operating at this time, since it is possible to leave these pumps in operation during the changing/insertion of a capillary gas chromatographic column. If, for some reason, the diffusion pumps and the mechanical pumps are not operating at this point, consult the Finnigan GC/MS Operations Manual for troubleshooting instructions and/or instructions for starting all of the pumps on the mass spectrometer system.

8.1 Procedure

8.1.1 To initiate operation of the vacuum system after the capillary column has been positioned in the gas chromatograph and both ends sealed, open the roughing valve toggle switch (red switch on top of the vacuum controller and temperature control panel). (Figure 7 presents an illustration of the vacuum controller panel of the mass spectrometer.)

8.1.2 As the mechanical pumps evacuate the ion source and analyzer regions of the mass spectrometer, monitor the forepressure in the ionizer by depressing the appropriate pushbutton on the vacuum controller panel.

8.1.3 When the ionizer forepressure reads 0.20 torr or lower, shut the red roughing valve and depress the baffle valve button (verify that the green dot is present) to open the ion source and analyzer regions of the mass spectrometer to diffusion pump vacuum.

8.1.4 When the ionizer forepressure reading is 0.01 torr or lower, depress the Ionization Gauge START pushbutton in order to light the ionization gauge and enable the Vacuum Protect circuitry.

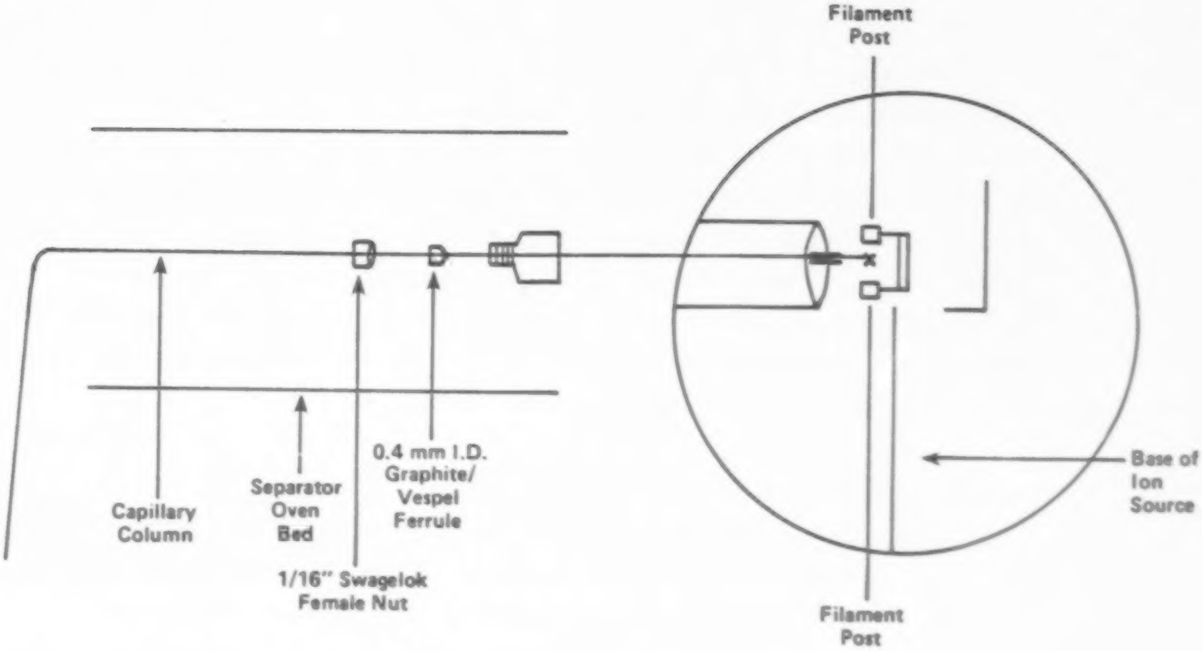

Filament Post
Capillary Column
Separator Oven Bed
1/16" Swagelok Female Nut
0.4 mm I.D. Graphite/ Vespel Ferrule
Base of Ion Source
Filament Post

Figure 6. Back end of GC column.

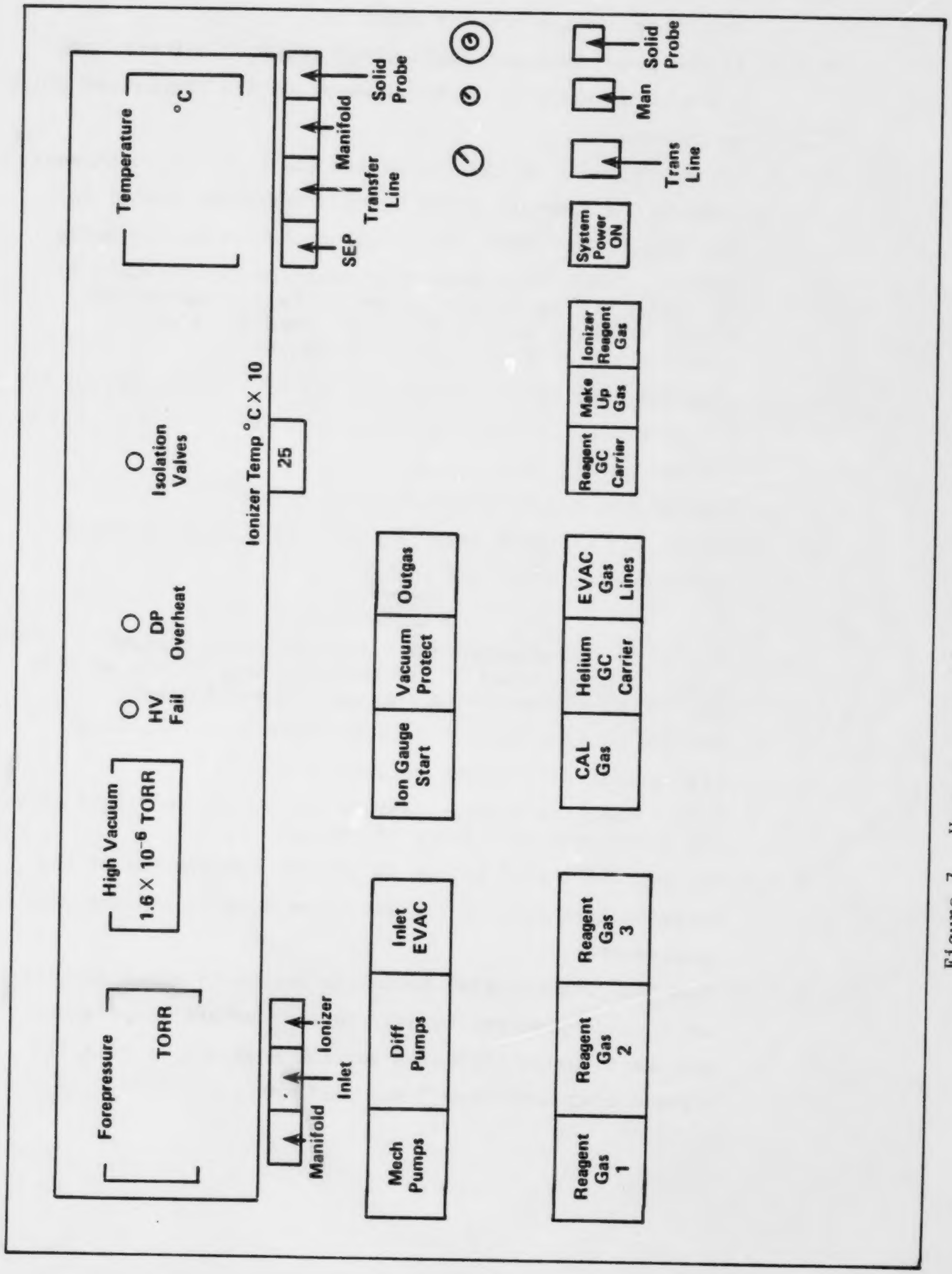

Forepressure
TORR
High Vacuum
1.6 × 10⁻⁶ TORR
HV Fail
DP Overheat
Isolation Valves
Temperature
°C
Ionizer Temp °C × 10
25
Manifold
Inlet
Ionizer
SEP
Transfer Line
Manifold
Solid Probe
Mech Pumps
Diff Pumps
Inlet EVAC
Ion Gauge Start
Vacuum Protect
Outgas
Reagent Gas 1
Reagent Gas 2
Reagent Gas 3
CAL Gas
Helium GC Carrier
EVAC Gas Lines
Reagent GC Carrier
Make Up Gas
Ionizer Reagent Gas
System Power ON
Trans Line
Man
Solid Probe

Figure 7. Vacuum controller and temperature controls.

8.1.5 If the ionization gauge does not light at this point, wait five minutes and try again.

8.1.6 If the gauge does not light after repeated efforts, use a meter to verify that the filament of the ionization gauge is continuous.

8.1.7 If the filament of the ionization gauge is not continuous, consult the Finnigan GC/MS Systems Operation Manual for the correct procedure for changing the ionization gauge.

NOTE: It will be necessary to vent the source/analyzer to change the ionization gauge, so follow the vacuum system start-up procedure as described above after installation of a new ionization gauge.

8.1.8 When the high vacuum reading is 3 x 10^{-5} torr, depress the Vacuum Protect pushbutton to allow power to be supplied to the Quadrupole Electronics.

8.1.9 Depress the Manifold Heater (MAN) pushbutton.

8.1.10 Adjust the manifold temperature to 92°C with the black potentiometer above the pushbutton.

CAUTION

During operation permanent damage to the secondary electron multiplier can occur if the manifold temperature exceeds 100°C with voltage going through the multiplier.

8.1.11 Set the Ionizer Temp °C x 10 thumbwheel control to 25 (equivalent to a direct reading of 250°C).

NOTE: When the ionizer reaches 250°C, the red light above the thumbwheel will flash on and off.

8.1.12 Depress TRANS LINE button to set the temperature of the transfer line between the gas chromatograph and the mass spectrometer.

8.1.13 Turn the potentiometer below the button to approximately the two o'clock position to set the appropriate temperature for the transfer line oven for the analysis of volatile organic compounds from Tenax cartridges.

8.1.14 Verify that the SEP, DIR, and CAP toggle switches are set to OFF, OFF, and GC CONTROL, respectively, on the Vacuum Diverter Panel (see Figure 8).

9.0 STARTING THE DUAL DISK DRIVES OF THE DATA GENERAL NOVE 3 COMPUTER

The Finnigan 4021 mass spectrometer and the Finnigan 3300 mass spectrometer share a common data system, the Data General Nova 3 computer with two Perkin-Elmer disk drives, each containing a removable and a fixed disk. It is therefore imperative that the operators on both systems establish clearly who will acquire data on which disk and what procedure will be followed if it is necessary for one of the operators to change one of the removable disks.

9.1 Procedure

9.1.1 Before starting the disk drives, check the thumbwheel dial on both the top and bottom disk drives.

9.1.2 Set the top drive at ZERO and the bottom drive at TWO in order to designate the removable disks as Units 0 and 2 while the two fixed disks are Units 1 and 3.

9.1.3 With disks present in both drives, depress the power switch on both drives.

9.1.4 Wait for the LOAD light to come on.

9.1.5 Be sure that the PROT CART and PROT FIXED switches on each drive are depressed in order to protect the disks against damage.

9.1.6 Flip the RUN/LOAD switch to RUN.

9.1.7 Wait for a READY light to appear on each drive.

9.1.8 When the READY light is observed, turn off the PROT CART and PROT FIXED switches; the disks are now ready for operation.

10. STARTING COMPUTER TERMINALS ON THE DATA GENERAL NOVA 3

The Finnigan 4021 and the Finnigan 3300 each have a Tektronix 4010 computer terminal which is routinely turned OFF at the end of the operating day.

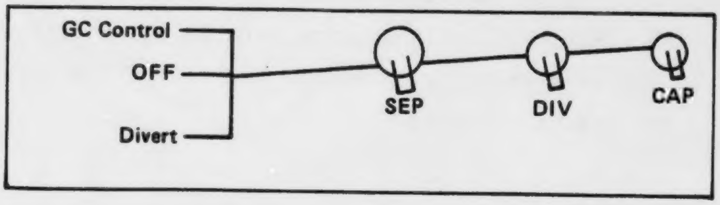

GC Control
OFF
Divert
SEP
DIV
CAP

Figure 8. Vacuum diverter panel.

10.1 Procedure

10.1.1 To prepare the terminal for operation, depress the MAIN POWER switch located beneath the terminal keyboard on a gray panel.

10.1.2 Verify that a red light labeled POWER on the keyboard panel lights when power is supplied to the terminal.

10.1.3 Check to be sure that the LOCAL/LINE switch on the terminal keyboard is in the LINE position.

11.0 STARTING THE FINNIGAN 4021 MASS SPECTROMETER

In routine operation, the mass spectrometer filament, electron multiplier, and Ionizer Mode switch on the Quadrupole Electronics Auxiliary Control Panel are turned OFF at the end of the operating day; all other temperatures and systems controls are left in operation.

11.1 Quadrupole Electronics Auxiliary Control Panel

11.1.1 Turn on the FILAMENT, MULTIPLIER, and IONIZER MODE switches on the Quadrupole Electronics Auxiliary Control Panel (see Figure 9).

11.1.2 Set the emission current of the filament at 0.5 mA; adjust the correct value with the knob labeled EMISSION CURRENT.

11.1.3 Turn the multiplier to the 2 kV position.

11.1.4 Adjust for the desired setting with the knob labeled FINE.

NOTE: The current setting for the multiplier is approximately 1750 volts, but the setting at any given time is a function of the age of the multiplier and other mass spectrometer system parameters.

11.1.5 Read the values for the emission current and the multiplier voltage by using the METER FUNCTION switch.

11.1.6 Set the IONIZER MODE switch to the EI/SEP position.

11.2 PPINICI Ion Control Module

11.2.1 On the PPINICI Ion Control Module (see Figure 10), set the DYNODE HV to the ON position, the IONIZER CONTROL to the QEM position, and the SCAN MODE to the POSITIVE setting.

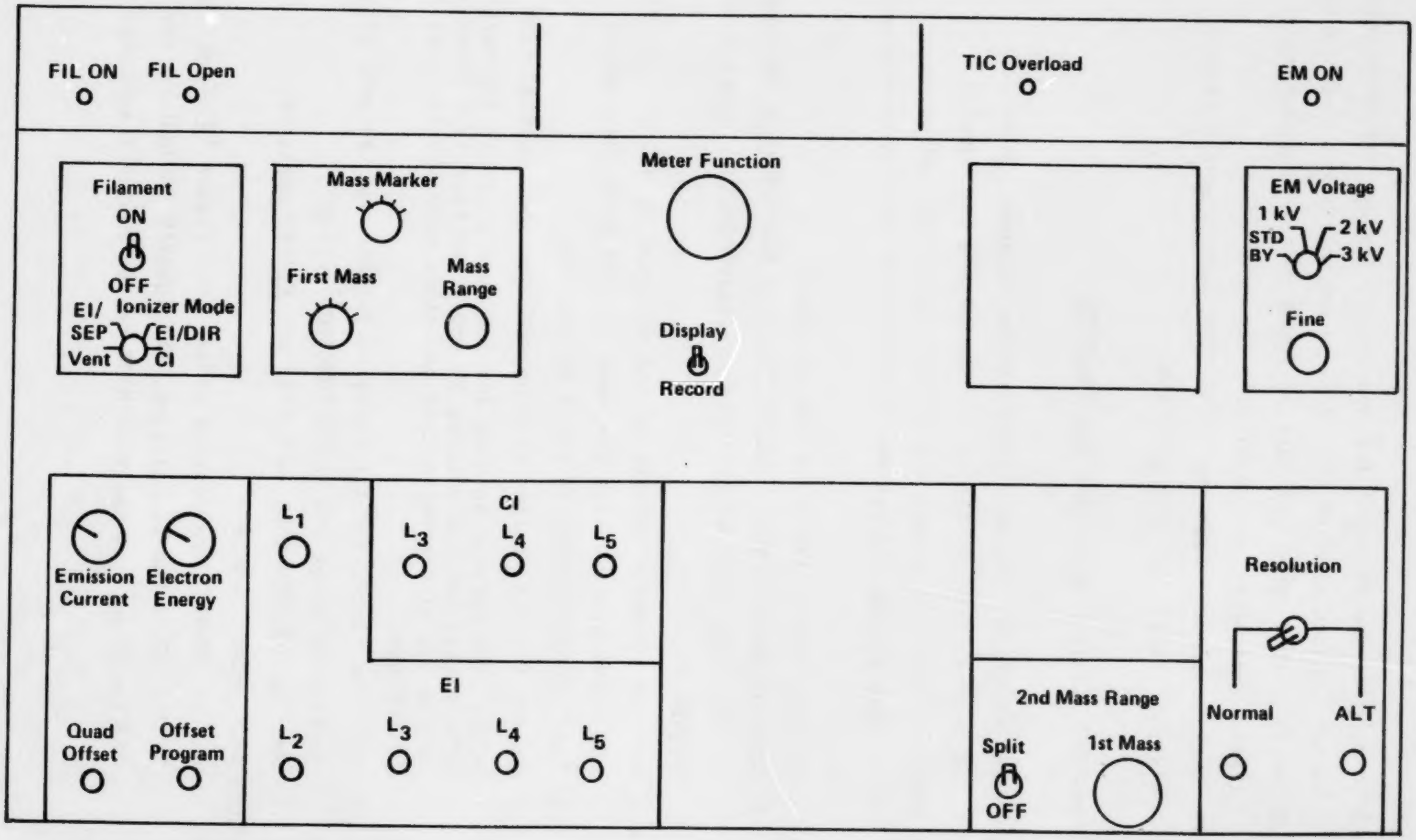

FIL ON
FIL Open
TIC Overload
EM ON
Filament
ON
OFF
EI/
SEP
Vent
Ionizer Mode
EI/DIR
CI
Mass Marker
First Mass
Mass
Range
Meter Function
Display
Record
EM Voltage
1 kV
STD
BY
2 kV
3 kV
Fine
Emission
Current
Electron
Energy
Quad
Offset
Offset
Program
L1
L2
CI
L3
L4
L5
EI
L3
L4
L5
2nd Mass Range
Split
OFF
1st Mass
Resolution
Normal
ALT

Figure 9. Quadrupole electronics auxiliary control panel.

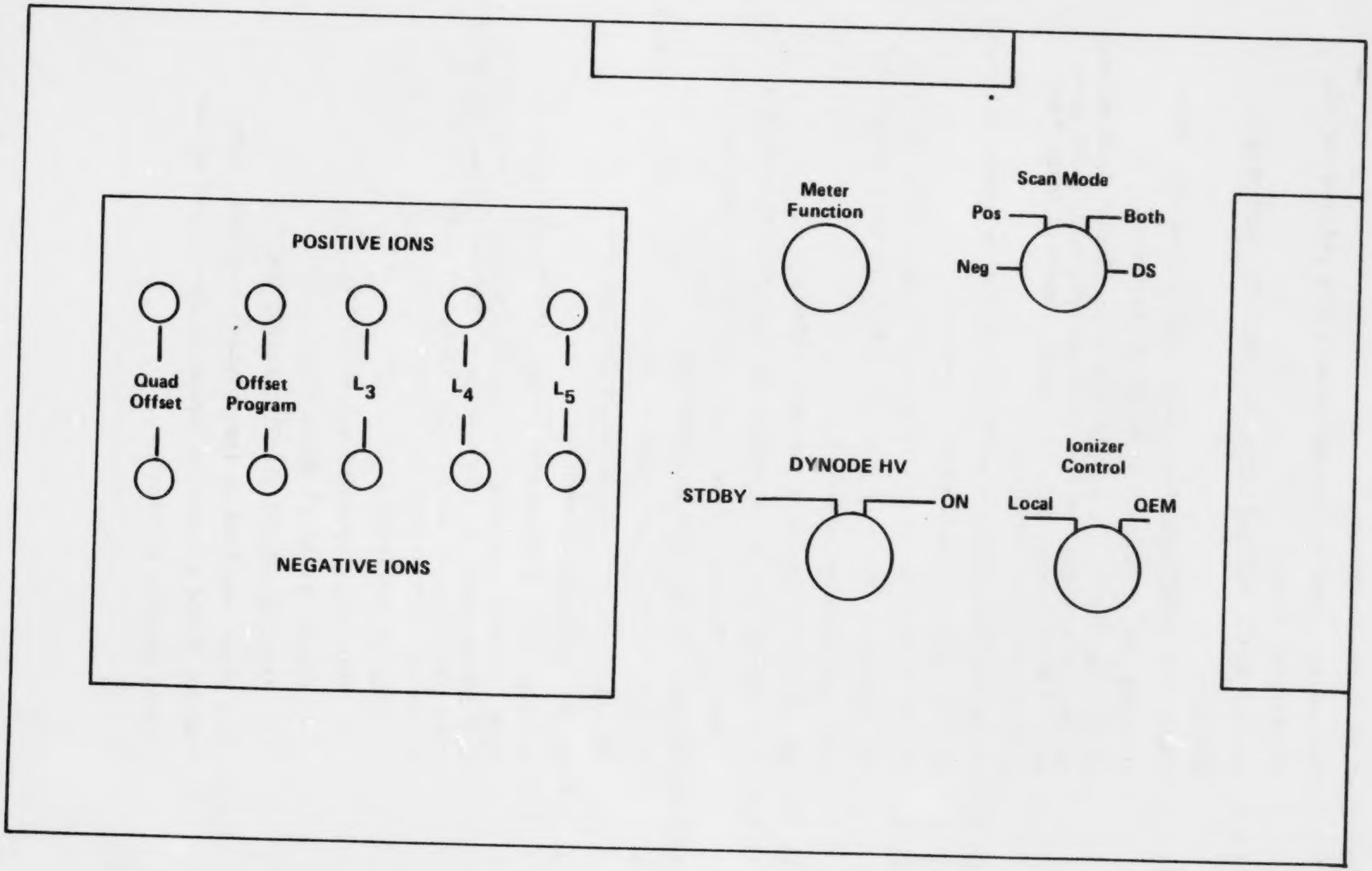

POSITIVE IONS
Quad Offset
Offset Program
L_3
L_4
L_5
NEGATIVE IONS
Meter Function
Scan Mode
Pos
Both
Neg
DS
DYNODE HV
STDBY
ON
Ionizer Control
Local
QEM

Figure 10. PPINICI ion control module.

11.3 Tuning of the Radio Frequency

11.3.1 Turn on the power to the oscilloscope by pulling out the cylindrical knob.

11.3.2 Set the SCOPE MODE switch to the MULTIPLE MASS MARKER position.

11.3.3 Rotate the FIRST MASS knob fully counterclockwise while rotating the MASS RANGE knob fully clockwise.

NOTE: The entire accessible mass range from 0-1000 daltons can now be observed on the oscilloscope, with small mass marks for each mass unit creating a somewhat fuzzy thick line on the oscilloscope.

11.3.4 Using the MASS MARKER and METER FUNCTION switches, determine at what mass the mass marks terminate.

11.3.5 If the mass marks do not terminate at mass 1000, adjust the Remote RF Assembly (the black box) located behind the mass spectrometer manifold.

11.3.6 Locate the RF Tuning Adjustment screw.

11.3.7 Turn the RF Tuning Adjustment Screw with a flat-bladed screwdriver until the multiple mass marks reach mass 1000.

11.4 Setting the Zero of the Mass Spectrometer

11.4.1 To zero the mass spectrometer, turn on the multiplier, dynode HV, and ionizer mode (EI/SEP position) switches.

11.4.2 Do not turn the filament on.

11.4.3 Set the SCOPE MODE switch in the DATA SYSTEM position.

NOTE: A computer procedure named ZF4 has been written to aid the operator in the zeroing of the mass spectrometer and this procedure is permanently stored on disk unit #1.
A copy of this procedure follows:

```
TRACE OF PROCEDURE ZF4
*  SCAN 4021(300;400;0.9;0;0;.1)
*  ACQU/O ZF4(K1;T1;M0;G1000;E)
*  MAP/C ZF4(G;360,370;V10;D1,1000,200)
```

11.4.4 With power supplied to the computer terminal, type a CONTROL D and a > should appear on the screen of the visual display terminal.

11.4.5 To activate the zeroing procedure type ZF4 followed by a carriage return <CR>.

NOTE: At this point the computer will show ions 360,362,...370 under the MAP/C program of the Finnigan Incos Data System.

11.4.6 Verify that random noise spikes are observed at these masses (see Figure 11).

11.4.7 If no noise spikes are observed or a great number appear, reset the zero of the mass spectrometer.

11.4.8 To regulate the number of the noise spikes, adjust the Zero Control knob located on the Sensitivity switch of the Output System front panel (see Figure 12).

11.4.9 When the MAP/C display produces random spikes at the mass displayed, type a Control D to regain operating control of the computer.

11.4.10 Type E <CR> to exit from the MAP/C program.

11.4.11 Terminate the ZF4 procedure by typing STOP <CR> when a caret (>) is observed.

12.0 INTRODUCTION OF PERFLUOROTRIBUTYLAMINE (FC-43) CALIBRATION GAS

In order to calibrate the mass spectrometer/computer system so that the correct masses will be assigned, it is necessary to introduce perfluorotributylamine (FC-43). This compound is routinely stored in a glass bulb attached to an inlet system on the far right flange of the manifold (see Figure 13).

12.1 Filling the Glass Bulb

12.1.1 If there is none of the calibration compound in the glass bulb, remove the bulb by loosening the female nut with a 9/16" open-end wrench.

12.1.2 Using a disposable pipet, fill the bulb with FC-43.

12.1.3 Remount the bulb on the inlet system.

12.1.4 Evacuate the FC-43 inlet line by opening the CAL GAS EVAC toggle valve for approximately ten seconds.

12.1.5 Depress the CAL GAS pushbutton on the Vacuum Controller panel.

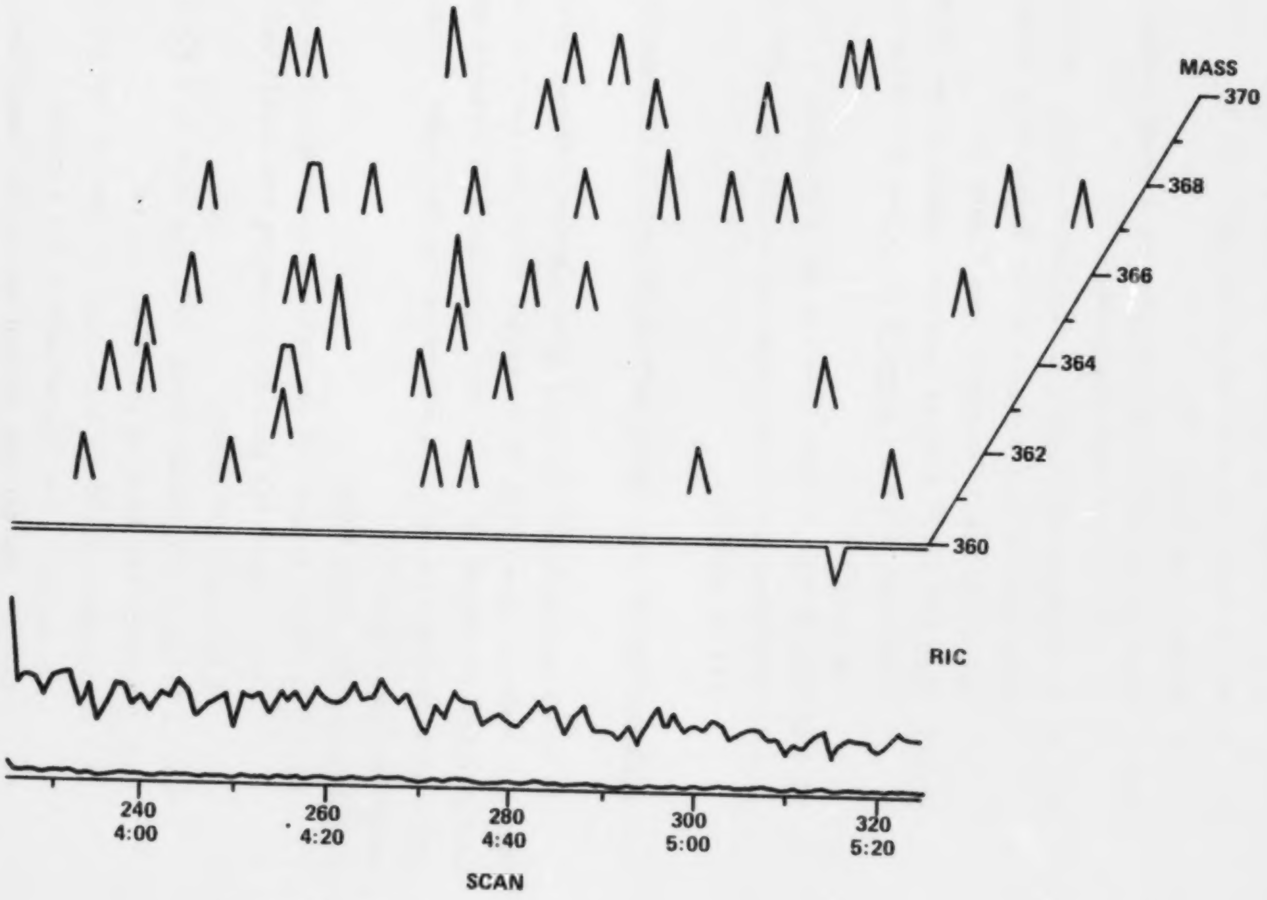


Figure 11. MAP/C with random spikes in ZMS.

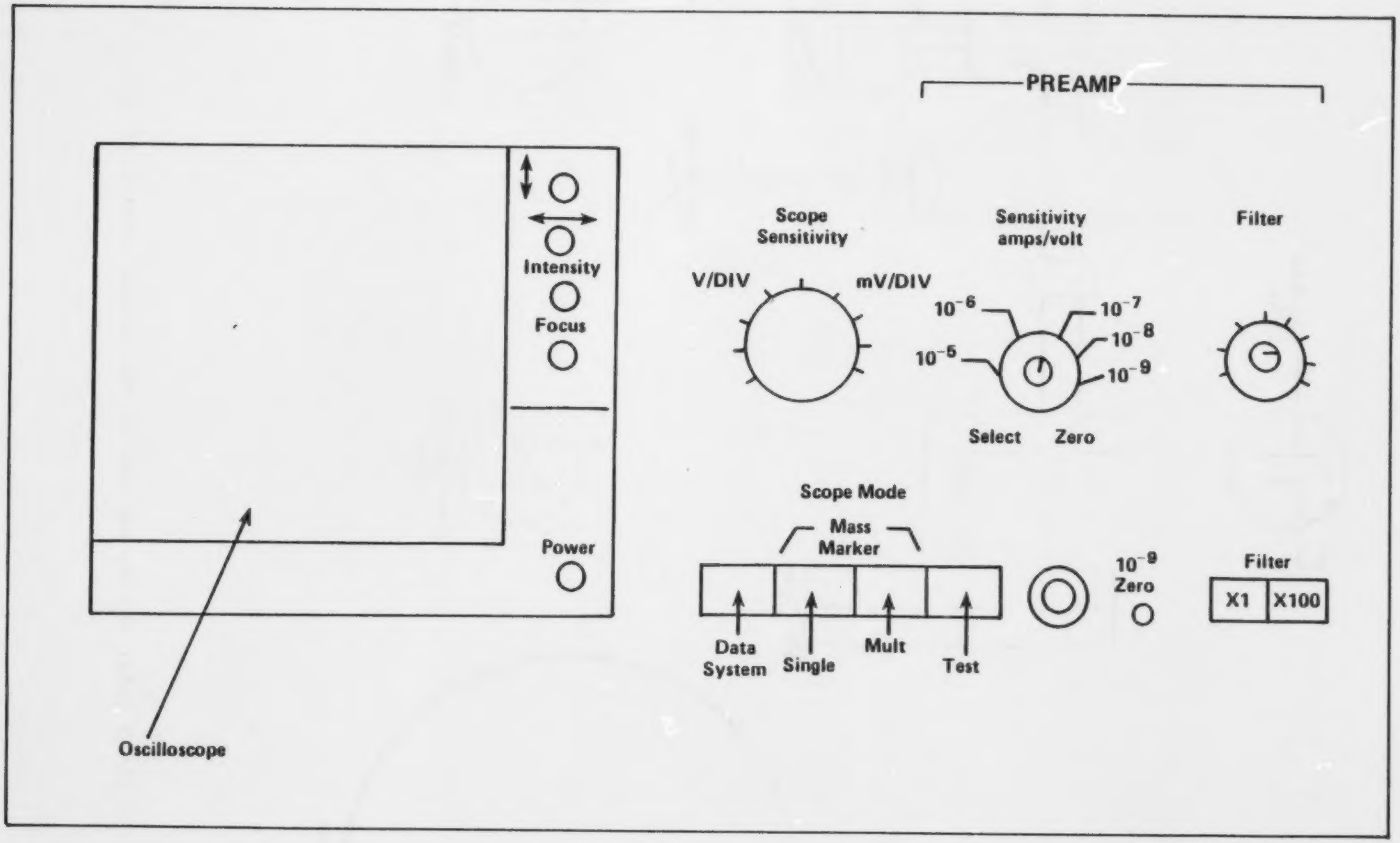

PREAMP
Scope Sensitivity
V/DIV
mV/DIV
Sensitivity amps/volt
10^{-6}
10^{-5}
10^{-7}
10^{-8}
10^{-9}
Select
Zero
Filter
Intensity
Focus
Power
Scope Mode
Mass Marker
Data System
Single
Mult
Test
10^{-9} Zero
Filter
X1
X100
Oscilloscope

Figure 12. Output system front panel.

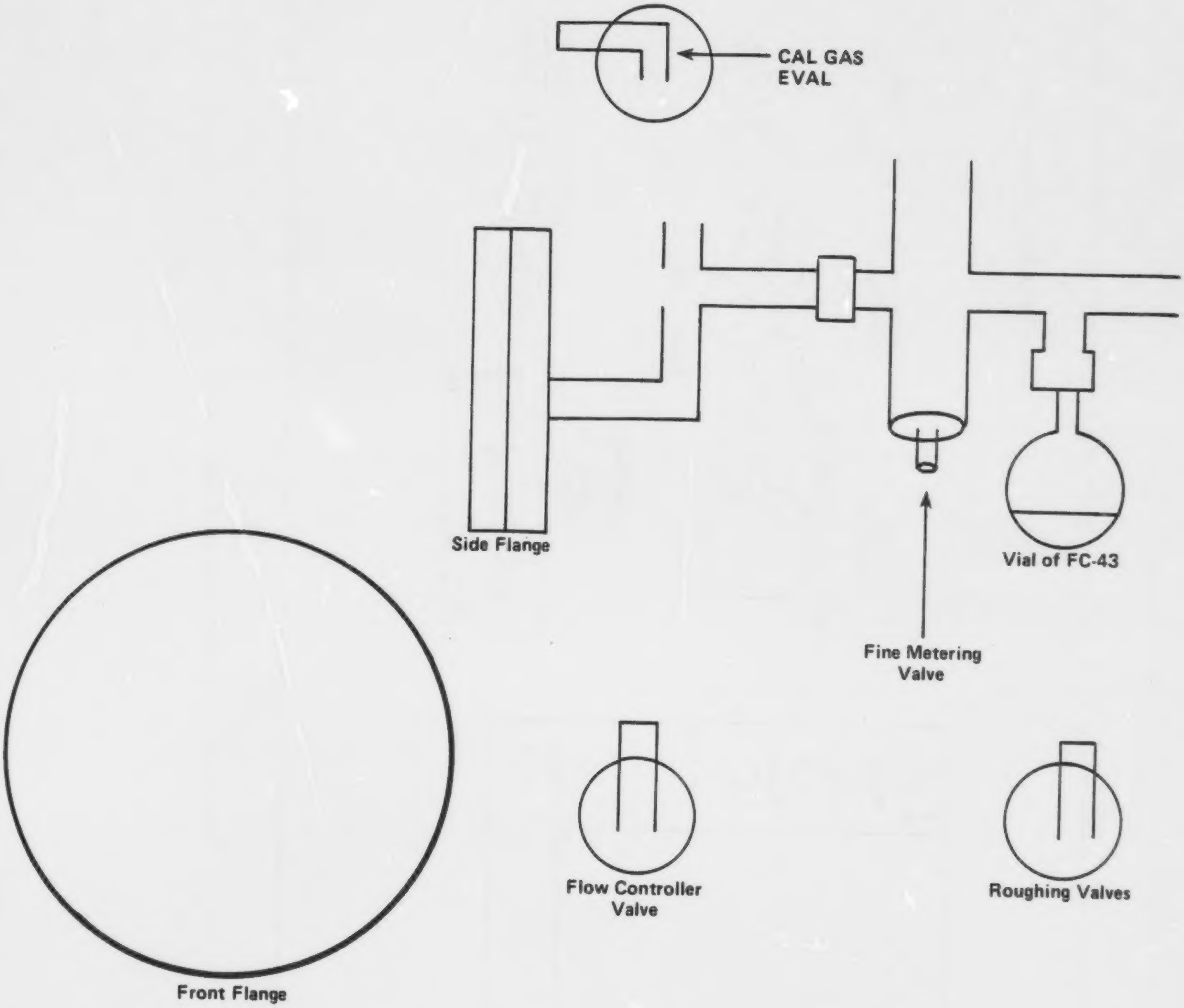

CAL GAS
EVAL
Side Flange
Vial of FC-43
Fine Metering
Valve
Front Flange
Flow Controller
Valve
Roughing Valves

Figure 13. FC-43 inlet system and top of the vacuum controller unit.

12.1.6 Slightly open the fine metering adjustment knob of the FC-43 gas inlet.

NOTE: If the FILAMENT, MULTIPLIER, IONIZER MODE, and DYNODE HV switches have been turned ON, ions produced by the FC-43 will be present on the screen of the oscilloscope.

12.1.7 Set the oscilloscope in the SINGLE MODE position.

13.0 TUNING THE MASS SPECTROMETER USING FC-43

The Finnigan 4021 mass spectrometer is tuned using a group of five lenses, an offset program, and a QUAD OFFSET control. These voltages must be adjusted to produce the greatest peak amplitude consistent with good peak shape. All controls pertinent to the tuning process are located on the Quadrupole Electronics Auxiliary Control Panel.

13.1 Procedure

13.1.1 Set the oscilloscope in SINGLE MODE.

13.1.2 Turn the OFFSET PROGRAM control fully counterclockwise.

13.1.3 Set the QUAD OFFSET potentiometer for 5 volts.

13.1.4 Set EI lens #1 and #3 for -10 volts.

13.1.5 Set EI lens #2 and #4 for -80 volts.

13.1.6 Set EI lens #5 for -20 volts.

13.1.7 Set the SPLIT/OFF switch for the second mass range to SPLIT.

13.1.8 Adjust the First Mass and Mass Range controls to center mass 100 on the left side of the oscilloscope.

13.1.9 Adjust the First Mass control of the second mass range to center mass 502 on the right side of the oscilloscope.

13.1.10 Adjust the QUAD OFFSET, lenses L1, L2, L3, and L4 controls for the greatest peak amplitude consistent with good peak shape.

13.1.11 Decrease the negative potential of lens #5 by turning the potentiometer counterclockwise to eliminate any liftoff of the front edge of the peak while maintaining good peak amplitude.

13.1.12 Fine-tune the ion source by making repeated alternating small readjustments to the QUAD OFFSET and LENS controls.

13.1.13 Adjust the OFFSET PROGRAM control for the greatest peak amplitude consistent with good peak shape at mass 502.

14.0 ACQUISITION OF DATA

The mass spectrometer is now ready to scan a mass spectrum of the calibration compound. The data system must now be prepared for the acquisition of data.

14.1 Setting Scan Parameters for the Acquisition of Data

NOTE: Before acquisition of data from the mass spectrometer can occur, it is essential to set the computer and interface to scan the appropriate mass range for the correct length of time.

14.1.1 In order to initiate computer-controlled scanning of the mass spectrometer, type SCAN 4021 <CR>.

NOTE: A series of questions will follow. The computer will have numerical values as the answers to these questions which will correspond to the last set of parameters used by the data system. These numerical values may or may not correspond to the values desired for calibration.

14.1.2 If the values presented by the computer are other than the values shown below, reset the values by typing the desired value after the value presented by the computer.

MASS RANGE (AMU)
LOW: 20 <CR>
HIGH: 650 <CR>
SCAN TIME (seconds)
UP: 0.95 <CR>
DOWN: 0.00 <CR>
HOLD TIME (seconds)
TOP: 0.00 <CR>
BOTTOM: 0.05 <CR>

NOTE: These scan parameters are used in all acquisition of data for instrument calibration using FC-43.

14.2 Setting Time Parameters for the Acquisition of Data

NOTE: Before acquisition of data can occur the computer clock must be set.

14.2.1 In order to set the computer clock type >TIME <CR>.

NOTE: A series of questions will follow. The computer will have no numerical values for any of these questions (unless time has already been set). The numerical value for the hour of day must be set as "army time."

14.2.2 Insert the numerical values between the limits the system specifies for month, day, year, hour, minute, and second.

NOTE: A value must be entered for each question; a <CR> is not adequate.

14.3 Initiation of Data Acquisition

14.3.1 Before initiating acquisition of data from the mass spectrometer, set the oscilloscope MODE switch to DATA SYSTEM.

14.3.2 Start the data acquisition by typing >ACQU disk#: file name, inst <CR>.

NOTE: For example, to initiate data acquisition on disk Unit #2 for file #4244 type >ACQU 2:4244,4021 <CR>. The program ACQU will then display a summary of the state of the data acquisition and if the computer is ready to acquire data, READY TO ACQUIRE will be observed in the top right position of the terminal screen.

14.3.3 Check the display of the summary to ensure that the scanning parameters, disk unit number, file names, and all other parameters are correct.

14.3.4 Type A <CR> to obtain the set of questions which allows the entry of accounting information and operator description of the data to be acquired.

14.3.5 To respond to the computer prompts for the questions, enter Sample Name, Operating Conditions, Compound Formula (if applicable), Compound Molecular Weight, Submitter, Account Number, and Analyst.

14.3.6 If a question does not apply for a particular analysis, type <CR> and the next question will be displayed.

NOTE: After the last question has been answered, the ACQU summary table will display all the entries made for the accounting information.

14.3.7 To initiate acquisition of data, type ACQU: G n <CR>, where n is the number of mass spectral scans desired.

14.4 Preparation of a Calibration Table

In order to obtain accurate mass assignments, the calibration table must be up-to-date before any analysis of samples is initiated. A calibration table is made by comparing time/intensity data acquired from scans of FC-43 with a mass/intensity reference table of FC-43 stored on disk Unit #1 on the Incos data system.

14.3.1 Acquire 15 to 20 scans of FC-43 to begin preparation of a calibration table.

14.4.2 After the acquisition of data is complete, type >CALI disk #: file name # scan number, reftable/modifiers <CR>.

NOTE: For example, >CALI 0:4244 #7, FC-43/E <CR> will calibrate the mass spectrometer/computer system using scan #7 of file 4244 compared to the FC-43 reference table and place the calibration table generated on disk unit #0.

14.4.3 Use the /E to eliminate certain background peaks which could affect the quality of the calibration.

14.4.4 Utilize the FIT program to measure the accuracy of the calibration.

14.4.5 Type >FIT <CR> to run the FIT program.

14.4.6 Examine the RMS error graph in the display generated by the FIT program to measure the accuracy of the mass assignments.

14.4.7 If the numerical value observed on the RMS error graph is greater than 50, repeat the FC-43 tuning procedure, perform another acquisition of data, and repeat the calibration process until the numerical value observed on the RMS error graph is less than 50.

14.4.8 Obtain a mass spectrum and list of the % relative abundances of the ions of FC-43 by typing >SPEC #n <CR>.

NOTE: For example: >SPEC #7 <CR> will display the seventh scan of the FC-43 data acquisition.

14.4.9 Print a hardcopy of the mass spectrum by typing SPEC: H <CR>.

14.4.10 Obtain a listing of the % relative abundances of all of the ions of the mass spectrum by typing SPEC: L <CR>.

14.4.11 Generate a hardcopy of this list by typing LIST: H <CR>.

15.0 TUNING THE MASS SPECTROMETER TO MEET PERFORMANCE SPECIFICATIONS FOR OCTAFLUOROTOLUENE (PFT)

Control of the percent relative abundances of ions of perfluorotoluene, the compound selected as the tuning standard for the analysis of volatile organic compounds from Tenax cartridges, is essential for obtaining data of the desired quality. The relative abundances of the ions of perfluorotoluene should be reproducible within a specific range established by a historical data base from day-to-day. Tuning criteria set for the major ions of the mass spectrum of PFT are as follows:

Ion	% RA of Base Peak	CV
69	29	7
79	7	3
93	15	4
117	39	9
167	12	2
186	59	6
217	100	0
236	75	8

Ideally the % RA should not vary by more than 10%.

15.1 Verifying the Acceptability of the Spectrum Obtained for Perfluorotoluene

NOTE: The initial tuning of the Finnigan 4021 mass spectrometer to the manufacturer's criteria for an acceptable tune for the calibration compound, FC-43, does not guarantee that an acceptable mass spectrum for PFT will be obtained.

15.1.1 Check the appearance of the mass spectrum of PFT by introducing PFT into the ion source of the mass spectrometer.

15.1.2 Replace the glass bulb used for FC-43 on the calibration gas inlet system with a bulb filled with PFT.

15.1.3 Introduce PFT to the ion source by the same procedure used for introduction of FC-43 (see Figure 13).

15.1.4 When PFT is present in the ion source, set the oscilloscope in SINGLE mode and set the First Mass Control for mass 50 and the Last Mass for mass 250.

15.1.5 Observe all the major ions of the PFT mass spectrum on the oscilloscope.

15.1.6 Set up data acquisition and acquire several scans of the PFT mass spectrum.

15.1.7 Select one scan to check the % RA.

NOTE: The spectrum of PFT obtained by introduction of PFT into the ion source using the calibration gas inlet will differ slightly from the PFT mass spectrum obtained by thermal desorption of the PFT from a Tenax cartridge and introduction to the mass spectrometer through the GC column, but the appearance of the mass spectrum should be a very good indication of whether the tune will meet the criteria for acceptable analysis.

15.2 Adjustment of the Tune of the Mass Spectrometer to Meet PFT Tuning Criteria

15.2.1 If the mass spectrum of PFT does not meet the criteria for instrument operation, retune the instrument to meet these criteria.

15.2.2 Adjust the five EI lenses, Quad Offset, and Offset Program voltages until the desired spectrum is obtained.

15.2.3 After any given series of adjustments, set up an acquisition and select a scan of PFT to check the tune.

15.2.4 Repeat the adjustment/acquisition process as many times as necessary in order to obtain an acceptable mass spectrum of PFT.

NOTE: Remember that in tuning for an acceptable mass spectrum of PFT, low mass ions such as 69 and 117 will be approximately 25% lower when observed from the thermal desorption process than when observed after introduction from the calibration gas inlet. For example, if a relative abundance of 40% for mass 69 is obtained when PFT is introduced through the calibration gas inlet, the relative abundance of this ion will be no greater than 30% when PFT is introduced to the mass spectrometer by thermal desorption. Ions of higher mass such as 186, 217, and 236 are not affected very much by the change in inlet systems. Therefore, in order to meet PFT tuning specifications when the compound is introduced by thermal desorption, the %RA of PFT ions obtained when the compound is introduced through the calibration gas inlet should be approximately the following:

Ion	% RA
69	40
79	8
93	18
117	50
167	16
186	65
217	100
236	75

16.0 ESTABLISHMENT OF OPERATING CONDITIONS FOR THE GAS CHROMATOGRAPH

The Finnigan 4021 gas chromatograph/mass spectrometer/data system has a Finnigan 9610 Gas Chromatograph (GC) which operates through a microprocessor to control temperatures and GC programming conditions.

16.1 Adjustment of Transfer Line Oven and Injector Block Temperatures

16.1.1 Set the temperature of the transfer line oven at 250°C by depressing the DET B TEMP button of the microprocessor, the numbers 250, and the ENTER key.

16.1.2 To set the injector block to 250°C, depress INJ TEMP, the numbers 250, and ENTER.

16.1.3 Verify that these temperatures have been entered accurately by recalling them through the microprocessor: depress DET B TEMP, then the RECALL key, and INJ TEMP and the RECALL key.

16.1.4 Monitor the temperature of the transfer line oven and the injector block by watching the GC Parameter Indicator Panel of the microprocessor: a red indicator light under the appropriate parameter indicates the status of that parameter.

NOTE: If the actual temperature is greater than the value entered into the microprocessor, the upper indicator light will be lit. When the temperature is lower than the value stored in the microprocessor the lower indicator will be lit.

16.2 Methods to Program the Gas Chromatograph

The temperature programming of the gas chromatograph is also controlled through the microprocessor. The desired initial gas chromatograph oven temperature for thermal desorption is 30°C, which should be held for five minutes. An initial temperature of 30°C is not entered into the microprocessor because, under normal operating conditions, it is impossible for the oven of the gas chromatograph to attain a temperature of 30°C and stabilize at this temperature.

16.2.1 Store a gas chromatograph temperature program of 45°C for 5 minutes then 4°C/min to 240°C in the microprocessor by using a method and three sequence controls.

16.2.2 Use the first sequence for initial column temperature and hold time.

16.2.3 Use the second sequence for final column temperature and rate of programming while utilizing the third sequence for final column temperature and hold time.

16.2.4 To enter 45°C as the initial temperature with a five minute hold, depress METHOD 1 SEQUENCE 1 COL TEMP 45 ENTER TIME 5.0 ENTER (see Figure 14).

16.2.5 Depress METHOD 1 SEQUENCE 2 COL TEMP 240 ENTER RATE 4.0 ENTER.

16.2.6 Type METHOD 1 SEQUENCE 3 COL TEMP 250 ENTER TIME 0.0 ENTER.

16.2.7 Enter a maximum column temperature value of 250°C by typing MAX TEMP 250 ENTER.

17.0 RECEIPT OF SAMPLES

17.1 Receive all Tenax cartridge tubes in a sealed can with the appropriate Custody Sheet after standards have been loaded on them.

17.2 Match the Custody Sheet with the corresponding sample to ensure no mixup has occurred.

17.3 Check each Custody Sheet carefully for the following items: 1) a signature of a person relinquishing custody; 2) the amounts of standards loaded on the cartridge; 3) the temperature and volume collected amounts; and 4) the Tenax batch number.

17.4 Do not analyze any sample that has no Custody Sheet or is missing any of the above information.

17.5 Put all cans of samples in the cartridge freezer when received.

17.6 Log each sample in the appropriate notebook as received.

17.7 Place each Custody Sheet in the project notebook (with all other information regarding that particular sample) after signing and dating it.

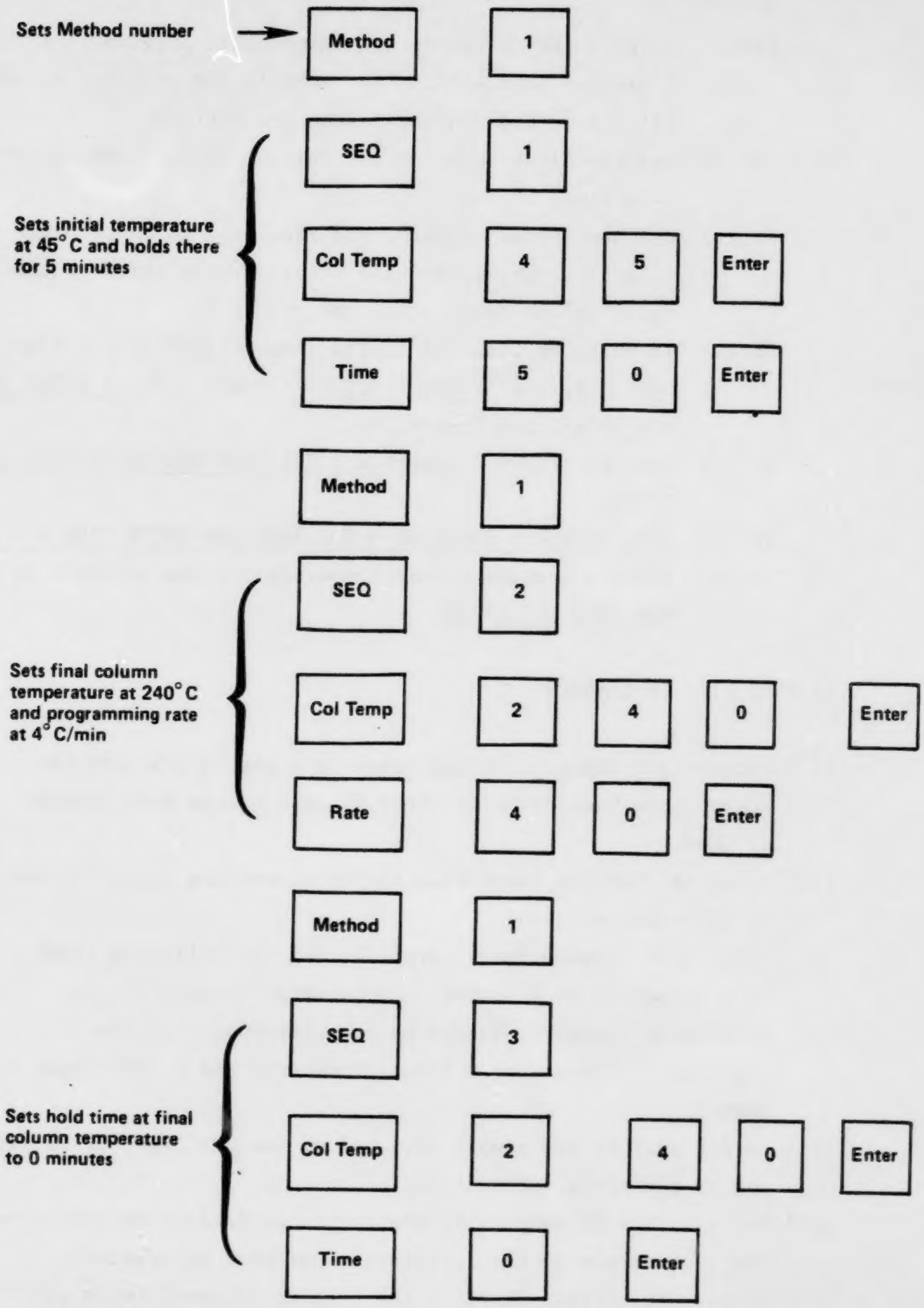

Sets Method number
Method
1
SEQ
1
Sets initial temperature at 45° C and holds there for 5 minutes
Col Temp
4
5
Enter
Time
5
0
Enter
Method
1
SEQ
2
Sets final column temperature at 240° C and programming rate at 4° C/min
Col Temp
2
4
0
Enter
Rate
4
0
Enter
Method
1
SEQ
3
Sets hold time at final column temperature to 0 minutes
Col Temp
2
4
0
Enter
Time
0
Enter

Figure 14. Example of temperature program for TEAM cartridges.

17.8 Store any used cartridges in sealed cans so they can be recycled, cleaned, and used again.

18.0 DESORPTION OF A TENAX CARTRIDGE

Thermal desorption of a Tenax cartridge is accomplished by heating the Tenax cartridge in the desorption chamber while purging helium gas through it. The volatile organic compounds desorbed are then collected in the nickel capillary trap which is cooled to liquid nitrogen temperatures. This trap is heated rapidly from liquid nitrogen temperatures to 250°C and the volatilized organic compounds are injected onto the gas chromatographic column which is directly coupled to the ion source of the mass spectrometer.

18.1 Desorption Procedure

18.1.1 To initiate the process of thermal desorption of a Tenax cartridge, open the toggle valve on the nitrogen gas line and pour liquid nitrogen into the Dewar flask which contains the coiled copper tubing of the nitrogen gas line.

18.1.2 Keep the Dewar flask full or nearly full in order to expedite the cooling process.

18.1.3 Fill the flask as often as necessary.

18.1.4 Adjust the knob of the Thermal Desorption Readout panel under the gas chromatograph to the second position in order to monitor the temperature of the trap.

18.1.5 Cool the oven of the gas chromatograph by typing METHOD 1 ENTER on the keyboard of the microprocessor.

18.1.6 Verify that the valve of the desorption unit is in the DESORB mode (up).

18.1.7 When the temperature reading for the trap reaches -190°C and liquid nitrogen is observed to drip at a slow but steady rate from the bottom of the trap, prepare a cartridge for the desorption process.

18.1.8 Remove the sealed paint can containing the desired cartridge from the freezer.

CAUTION

Use the freezer in the laboratory designated for cartridge storage ONLY for this purpose. Inadvertent storage of containers of solvent in this freezer will result in contamination of all cartridges stored in the freezer and will compromise the analysis, since organic solvents are frequently target compounds for quantitative analysis.

CAUTION

Verify that the laboratory personnel are not involved in any process which requires the presence of open containers of organic solvents as the fumes of organic solvents will hopelessly contaminate a Tenax cartridge exposed to this atmosphere for only a few seconds, thus compromising the quantitative and/or qualitative assay.

18.1.9 Open the sealed lid of the paint can, using a flat-bladed screwdriver, beverage can opener, or other convenient tool for this purpose.

NOTE: The cartridge will be in a glass culture tube with a Teflon-lined screw cap.

18.1.10 Remove a single culture tube from the paint can and place in the wooden cartridge holder in front of the gas chromatograph.

18.1.11 Seal the paint can and replace in the freezer.

18.1.12 Release the Teflon cap of the desorption chamber.

18.1.13 Remove the cartridge from the culture tube using forceps (do not touch a cartridge with your hands).

CAUTION

DO NOT TOUCH THE CARTRIDGE WITH YOUR HANDS! The slightest trace of organic compounds present on the fingertips can be sufficient to compromise the analysis. If the cartridge is inadvertently touched, make careful note of the circumstances in both the instrument log and the project notebook.

18.1.14 Insert the cartridge immediately into the desorption chamber.

18.1.15 Close the Teflon cap of the desorption chamber.

18.1.16 Initiate the timing of the eight-minute desorption cycle.

18.1.17 Check the flow of helium through the trap frequently to verify that the helium flow rate does not slow or stop, as problems with the gas flow at this point indicate that the moisture content of the cartridge is such that the trap is freezing.

18.1.18 If the helium flow slows or stops, note this observation both in the instrument log book and in the project notebook because the results of the analysis can be severely affected as a function of the exact point in the desorption cycle at which the freezing of the trap occurred.

CAUTION

Do not continue with the desorption of a Tenax cartridge if no helium purge flow is evident after a period of three to four minutes. No purge flow indicates the trap is frozen. Simply leave the valve in the DESORB mode and heat the trap to 240°C for approximately five minutes. Do not turn the valve to the INJECT position for this will allow a massive amount of water to bombard the ion source and cause the instrument to go into high vacuum failure. If the trap freezes in one minute or less, put the trap through three heating cycles in the DESORB mode before attempting to analyze another cartridge.

18.2 Injection Procedure

18.2.1 At the end of the eight-minute desorption cycle, close the nitrogen toggle valve.

18.2.2 Turn the desorption unit valve to the INJECT mode (down).

18.2.3 Initiate heating of the nickel trap.

18.2.4 Strike a CARRIAGE RETURN key on the terminal to begin acquisition of data.

18.2.5 Turn off the trap after it has heated to 240°C.

18.2.6 Shut the GC oven door five minutes after injection has occurred.

18.2.7 To start the GC program, type METHOD 1 ENTER START RUN on the microprocessor keyboard immediately after closing the door of the GC oven.

19.0 USE OF RESPONSE FACTORS IN THE QUANTITATION OF VOLATILE ORGANIC COMPOUNDS THERMALLY DESORBED FROM TENAX CARTRIDGES

Quantitation of volatile organic compounds is performed by the method of Response Factors. To determine a Response Factor, an analysis is performed of a cartridge containing known quantities of both the standard and the compound of interest. The Response Factor is calculated using the following equation:

RF = (peak area compound of interest/ng compound of interest)/(peak area standard/ng standard)

A set of two Tenax cartridges containing the standard and compounds of interest have been required in order to establish Response Factors for all of the compounds of interest for the TEAM study. The cartridges are designated as CPMX + RMR and RMR. The CPMX + RMR cartridges contain seven compounds used for determination of column performance parameters as well as target compounds which are loaded onto the cartridges via a Flash Evaporation System. RMR cartridges contain standard and other target compounds loaded via a Permeation System. A typical list of target compounds and number of nanograms/cartridge is presented below:

Compound	ng Loaded
Benzene	304
Chloroform	114
1,1,1-Trichloroethane	174
Carbon tetrachloride	201
1,2-Dichloroethane	329
Trichloroethylene	403
1,1,2,2-Tetrachloroethane	409
Chlorobenzene	140
Tetrachloroethylene	323
Ethylbenzene	346
p-Xylene	344
o-Xylene	352
Styrene	362
o-Dichlorobenzene	260
p-Dichlorobenzene	260
n-Octane	288
n-Decane	292
5-Nonanone*	328
Acetophenone*	411
2,6-Dimethylphenol*	294
2,6-Dimethylaniline*	391
1-Octanol*	330
Perfluorobenzene	125
Perfluorotoluene	130

*Used in the calculation of column performance parameters; not a target compound.

19.1 Establishment of a Response Factor Data Base

19.1.1 To establish a Response Factor data base for the target compounds prior to the analysis of field sample cartridges, analyze a series of five CPMX + RMR and five RMR cartridges by thermal desorption with GC/MS.

19.1.2 Calculate five sets of Response Factors for each ion of interest for each target compound.

19.1.3 Tabulate these Response Factors.

19.1.4 Calculate mean, standard deviation, and coefficient of variation.

19.1.5 If several Response Factors in a particular assay can be identified as outliers by their lack of correspondence to the other values obtained, discard that set of Response Factors.

19.1.6 Analyze another cartridge.

19.1.7 If the set of five Response Factors does not appear to be consistent, immediately check the desorption unit, gas chromatograph, and mass spectrometer for the presence of an air leak or some other problem, rectify the problem, and repeat the series of five cartridges.

19.2 Quantitation of Response Factors for Target Compounds

19.2.1 Initiate quantitation of Response Factors for target compounds by creating a Procedure in the Incos data system to obtain ion peak areas and scan numbers automatically for each ion of each compound.

NOTE: A procedure is simply a string of commands concatenated to make data processing more efficient. The procedure also incorporates a Method file which acts like a smaller Procedure.

An example of a Procedure named TEST for ions 117 and 186 of Perfluorobenzene and the 61, 97, and 99 ions of 1,1,1-Trichloroethane can be entered on the computer terminal as follows:

```
>TEST  =  SET Q $ <CR>
       =  EDQL (-; W; E) <CR>
       =  CHRO (I; R; 186; 117; X291; @RP; E) <CR>
       =  CHRO (I; R; 61; 97; 99; X334; @RP; E) <CR>
       =  QUAN (I; F1; H; E) <CR>
       =  FEED <CR>
       =  BEEP <CR>
       =  <CR>
```

19.2.2 Enter the Method file into the computer separately and designate the Method file by its own name.

NOTE: For example, Method file RP is entered in the following way:

```
>CHRORP = G-25,35;N-2,2;A>-2,2;D-25,45; <CR> <CR>
```

The Procedure includes information about compound ions, the scan number at which the compound should be sought, and the Method file. The Method file contains information needed to designate a mass range in which to search for ions of the target compounds and establishes parameters required for peak area quantitation. PERF 1 and CRMR are the Procedure names currently being used for RMR and CPMX + RMR quantitation. A trace of each of the procedures is presented below:

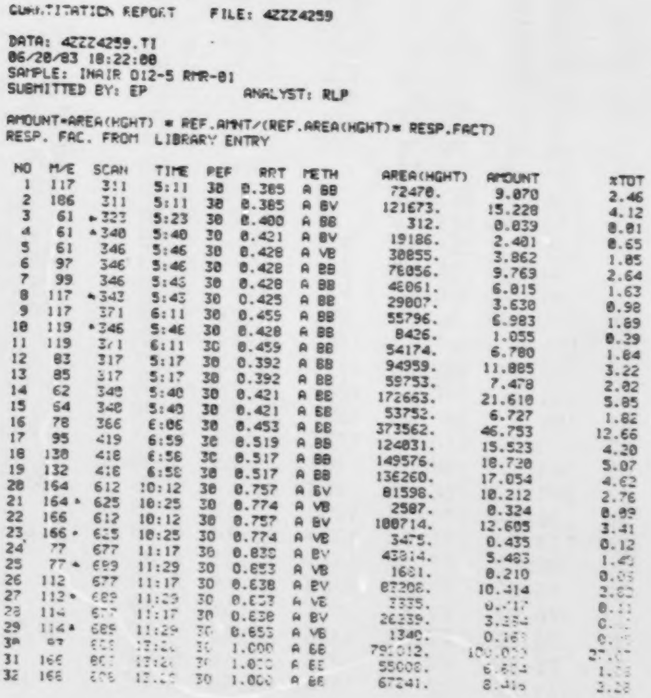
```
TRACE OF PROCEDURE PERF 1

* SETQ $
* EDQL (-;W;E)
* CHRO(I;R;186;117;X291;@RP;E)
* CHRO(I;R;61;97;99;X334;@RP;E)
* CHRO(I;R;117;119;X359;@RP;E)
* CHRO(I;R;83;85;X300;@RP;E)
* CHRO(I;R;62;64;X328;@RP;E)
* CHRO(I;R;78;X354;@RP;E)
* CHRO(I;R;95;130;132;X411;@RP;E)
* CHRO(I;R;164;166;X624;@RP;E)
* CHRO(I;R;77;112;114;X688;@RP;E)
* CHRO(I;R;83;166;168/X814;@RP;E)
* QUAN(I;F1;H;E)
* FEED
* BEEP
```

```
TRACE OF PROCEDURE CRMR
* SETQ $
* EDQL (-;W;E)
* CHRO(I;R;186;117;X294;@RP;E)
* CHRO(I;R;104;X790;@RP;E)
* CHRO(I;R;146;148;X1067;@RP;E)
* CHRO(I;R;146;148;X1119;@RP;E)
* CHRO(I;R;91;106;X729;@RP;E)
* CHRO(I;R;91;106;X751;@RP;E)
* CHRO(I;R;91;106;X801;@RP;E)
* QUAN(I;F1;H;E)
* FEED
* BEEP
```

19.2.3 To use the PERF1 Procedure after analyzing an RMR cartridge, simply type >PERF1 <CR>.

NOTE: The computer automatically obtains quantitation data and prints a hardcopy of the quantitation data on the Versatec printer. An example of a quantitated RMR cartridge using Procedure PERF1 is presented below:



```
QUANTITATION REPORT    FILE: 42Z24259

DATA: 42Z24259.TI
06/20/83 10:22:00
SAMPLE: INAIR 012-5 RMR-01
SUBMITTED BY: EP              ANALYST: RLP

AMOUNT=AREA(HGHT) * REF.AMNT/(REF.AREA(HGHT)* RESP.FACT)
RESP. FAC. FROM  LIBRARY ENTRY

 NO  M/E   SCAN   TIME  REF   RRT   METH    AREA(HGHT)  AMOUNT         %TOT
  1  117    311   5:11  30  0.385   A BB      72470.     9.070         2.46
  2  186    311   5:11  30  0.385   A BV     121673.    15.228         4.12
  3   61  * 323   5:23  30  0.400   A BB        312.     0.039         0.01
  4   61  * 340   5:40  30  0.421   A BV      19186.     2.401         0.65
  5   61    346   5:46  30  0.428   A VB      30855.     3.862         1.05
  6   97    346   5:46  30  0.428   A BB      78056.     9.769         2.64
  7   99    346   5:46  30  0.428   A BB      48061.     6.015         1.63
  8  117  * 343   5:43  30  0.425   A BB      29007.     3.630         0.98
  9  117    371   6:11  30  0.459   A BB      55796.     6.983         1.89
 10  119  * 346   5:46  30  0.428   A BB       8426.     1.055         0.29
 11  119    371   6:11  30  0.459   A BB      54174.     6.780         1.84
 12   83    317   5:17  30  0.392   A BB      94959.    11.885         3.22
 13   85    317   5:17  30  0.392   A BB      59753.     7.478         2.02
 14   62    340   5:40  30  0.421   A BB     172663.    21.610         5.85
 15   64    340   5:40  30  0.421   A BB      53752.     6.727         1.82
 16   78    366   6:06  30  0.453   A BB     373562.    46.753        12.66
 17   95    419   6:59  30  0.519   A BB     124031.    15.523         4.20
 18  130    418   6:58  30  0.517   A BB     149576.    18.720         5.07
 19  132    418   6:58  30  0.517   A BB     136260.    17.054         4.62
 20  164    612  10:12  30  0.757   A BV      81598.    10.212         2.76
 21  164 *  625  10:25  30  0.774   A VB       2587.     0.324         0.09
 22  166    612  10:12  30  0.757   A BV     100714.    12.605         3.41
 23  166 *  625  10:25  30  0.774   A VB       3475.     0.435         0.12
 24   77    677  11:17  30  0.830   A BV      43814.     5.483         1.4[illegible]
 25   77 *  689  11:29  30  0.853   A VB       1681.     0.210         0.0[illegible]
 26  112    677  11:17  30  0.838   A BV      83208.    10.414         2.8[illegible]
 27  112 *  689  11:29  30  0.853   A VB       3335.     0.417         0.1[illegible]
 28  114    677  11:17  30  0.838   A BV      26239.     3.284         0.[illegible]
 29  114 *  689  11:29  30  0.853   A VB       1340.     0.168         0.[illegible]
 30  [illegible] [illegible] [illegible]  30  1.000   A BB     79[illegible]012.   100.000        27.[illegible]
 31  166  [illegible] [illegible]  30  1.000   A BB      55008.     6.8[illegible]         1.[illegible]
 32  166  [illegible] [illegible]  30  1.000   A BB      67241.     8.415         2.28
```

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In the computer output presented above, some of the ions had asterisks beside them. These asterisks were inserted by the operator to indicate that some extraneous ions were quantitated by the computer which do not belong to a target compound. These extraneous ions were found in the same scan range and ion channel being monitored. All of these extraneous ions must be deleted to avoid reporting misleading and/or false data.

19.3 Deletion of Extraneous Ions from a Quantitation List

19.3.1 Use a program called the Quantitation List Editor (EDQL) to make deletions of extraneous ions.

19.3.2 Enter the EDQL program by typing >EDQL file name <CR>.

NOTE: A printout similar to that obtained from the QUAN program will be observed:

```
REFERENCE RETENTION TIME (MIN:SEC)    24:40
 NO. LOW MASS   HIGH   SCAN    TIME  METH         AREA    HEIGHT   NAME NUM
  1   116.54  117.54    562    9:22  A BB       53302.    10752.   --     0
  2   185.56  186.56    562    9:22  A BB       90503.    18267.   --     0
  3    82.53   83.53    584    9:44  A BB       29169.     5004.   --     0
  4    84.53   85.53    584    9:44  A BB       20439.     3274.   --     0
  5   185.56  186.56    649   10:49  A BB       61539.    10937.   --     0
  6   235.57  236.57    649   10:49  A BB       69235.    11860.   --     0
  7    63.52   64.52    652   10:52  A BB         819.      170.   --     0
  8    60.52   61.52    676   11:16  A BB      158133.    30524.   --     0
  9    96.53   97.53    676   11:16  A BB      333006.    62839.   --     0
 10    98.53   99.53    676   11:16  A BB      217480.    41204.   --     0
 11    77.52   78.52    729   12:09  A BB      467079.    75753.   --     0
 12   116.54  117.54    745   12:25  A BB        5810.     1140.   --     0
 13   118.54  119.54    745   12:25  A BB        5734.     1118.   --     0
 14   128.54  129.54    856   14:16  A BB        1206.      282.   --     0
 15    94.53   95.53    861   14:21  A BV       63520.    13767.   --     0
 16   129.54  130.54    861   14:21  A BB       73057.    15771.   --     0
 17   131.54  132.54    861   14:21  A BB       70061.    14776.   --     0
 18   126.54  127.54   1173   19:33  A BB        6049.     1384.   --     0
 19   128.54  129.54   1173   19:33  A BB        7759.     1768.   --     0
 20   106.53  107.53   1209   20:09  A BB         163.       53.   --     0
 21   108.53  109.53   1209   20:09  A BB          90.       32.   --     0
 22   113.53  114.53   1276   21:16  A BB        7941.     2272.   --     0
 23   163.55  164.55   1283   21:23  A BB      113100.    23967.   --     0
 24   165.55  166.55   1283   21:23  A BB      142233.    30271.   --     0
 25   130.54  131.54   1355   22:35  A BB         282.       85.   --     0
 26   132.54  133.54   1354   22:34  A BB        6917.     2028.   --     0
 27    76.52   77.52   1365   22:45  A BB          13.        7.   --     0
 28    76.52   77.52   1368   22:48  A BB          46.       12.   --     0
 29    76.52   77.52   1375   22:55  A BB          86.       28.   --     0
 30    76.52   77.52   1385   23:05  A BB        7681.     1694.   --     0
 31    76.52   77.52   1398   23:18  A BB          79.       23.   --     0
 32   111.53  112.53   1376   22:56  A BV        1957.      437.   --     0
```

19.3.3 Make all deletions from the bottom to the top of the list in EDQL to avoid having entry numbers changed by the computer and thus making incorrect deletions.

19.3.4 From inspection of the Quantitation Report, one sees asterisks at numbers 28, 27, 25, 23, 21, 10, 8, 4 and 3.

19.3.5 In the EDQL program, type EDQL: -28; -27; -25; -23; -21; -10; -8; -4; -3 <CR> and a revised quantitation list with no extraneous ions will appear.

19.3.6 Type EDQL: W <CR> to save all of these data and write the data to an output file for future use.

NOTE: The updated EDQL list is presented below:

```
REFERENCE RETENTION TIME (MIN:SEC)   24:40
 NO. LOW MASS  HIGH  SCAN   TIME  METH         AREA   HEIGHT   NAME NUM
   1  116.54  117.54  562   9:22  A BB        53302.   10752.  --     0
   2  185.56  186.56  562   9:22  A BB        90503.   18267.  --     0
   3   82.53   83.53  584   9:44  A BB        29169.    5004.  --     0
   4   84.53   85.53  584   9:44  A BB        20439.    3274.  --     0
   5  185.56  186.56  649  10:49  A BB        61539.   10937.  --     0
   6  235.57  236.57  649  10:49  A BB        69235.   11860.  --     0
   7   63.52   64.52  652  10:52  A BB          819.     170.  --     0
   8   60.52   61.52  676  11:16  A BB       158133.   30524.  --     0
   9   96.53   97.53  676  11:16  A BB       333006.   62839.  --     0
  10   98.53   99.53  676  11:16  A BB       217480.   41204.  --     0
  11   77.32   78.52  729  12:09  A BB       467079.   75703.  --     0
  12  116.54  117.54  745  12:25  A BB         5810.    1140.  --     0
  13  118.54  119.54  745  12:25  A BB         5734.    1118.  --     0
  14  128.54  129.54  856  14:16  A BB         1206.     282.  --     0
  15   94.53   95.53  861  14:21  A BV        63520.   13767.  --     0
  16  129.54  130.54  861  14:21  A BB        73057.   15771.  --     0
  17  131.54  132.54  861  14:21  A BB        70061.   14776.  --     0
  18  126.54  127.54 1173  19:33  A BB         6049.    1384.  --     0
  19  128.54  129.54 1173  19:33  A BB         7759.    1768.  --     0
  20  106.53  107.53 1209  20:09  A BB          163.      53.  --     0
  21  108.53  109.53 1209  20:09  A BB           90.      32.  --     0
  22  113.53  114.53 1276  21:16  A BB         7941.    2272.  --     0
  23  163.55  164.55 1283  21:23  A BB       113100.   23967.  --     0
  24  165.55  166.55 1283  21:23  A BB       142233.   30271.  --     0
  25  113.53  114.53 1385  23:05  A BB         4659.    1076.  --     0
  26   90.53   91.53 1451  24:11  A BB       350865.   77882.  --     0
  27  105.53  106.53 1451  24:11  A BB       124249.   28106.  --     0
  28  172.55  173.55 1481  24:41  A BB          939.     182.  --     0
  29   90.53   91.53 1480  24:40  A BB       679212.   88403.  --     0
  30  105.53  106.53 1481  24:41  A BB       426767.   60853.  --     0
  31  103.53  104.53 1538  25:38  A BV       191381.   43840.  --     0
  32   90.53   91.53 1555  25:55  A BV       264121.   59714.  --     0
  33  105.53  106.53 1555  25:55  A BV       149688.   34543.  --     0
  34  120.54  121.54 1723  28:43  A BB        36983.    8648.  --     0
  35  135.54  136.54 1723  28:43  A BB        30142.    7024.  --     0
  36  145.54  146.54 1887  31:27  A BB        10122.    2724.  --     0
  37  145.54  146.54 1901  31:41  A BB        82268.   18496.  --     0
  38  147.54  148.54 1887  31:27  A BB         6477.    1750.  --     0
  39  147.54  148.54 1901  31:41  A BB        52232.   11712.  --     0
  40  141.54  142.54 1907  31:47  A BB        14904.    4060.  --     0
  41  145.54  146.54 1966  32:46  A BB        14316.    3608.  --     0
  42  147.54  148.54 1966  32:46  A BB         8990.    2276.  --     0
  43  155.55  156.55 2172  36:12  A BB        13501.    3516.  --     0
  44  169.55  170.55 2416  40:16  A BB         6968.    1884.  --     0
```

19.4 Creation of Libraries for CPMX + RMR and RMR Compounds

The next step in calculation of Response Factors is creation of appropriate libraries for the target compounds.

19.4.1 Designate each library by a two-letter name.

19.4.2 Do not attempt to create more than one library at a time.

19.4.3 Before entry of a specific compound in the library, list the library name under SET4 of the STAT program.

NOTE: The library in current use for the RMR target compounds is designated library RR.

19.4.4 To enter library name RR in SET4 under the STAT program type >STAT <CR> followed by >SET4 RR <CR>.

19.4.5 Set a mass spectrum for each compound in library RR.

19.4.6 Obtain a scan number for each compound from the Quantitation List from an RMR cartridge analysis.

19.4.7 Use the scan number for a compound under the CHRO program for a library search.

19.4.8 Enter CHRO by typing >CHRO file name, cali table <CR>.

19.4.9 Be sure that the standard compound ion to be used as the quantitation standard is the first entry in a library.

NOTE: Since the ion of mass 186 of perfluorobenzene is this ion, make it the first entry of library RR.

19.4.10 If Scan #260 corresponds to the elution position of perfluorobenzene (PFB), type CHRO: S260;Y <CR>.

19.4.11 To enter the library editor program (EDLB) from the library search type LIBR: ED <CR>.

19.4.12 Type EDLB: I <CR> and answer the list of questions which follows:

NEW NAME: Type Perfluorobenzene <CR>
NEW FORMULA: Type C6 F6 <CR>
MOLECULAR WEIGHT: Type 186 <CR>
RETENTION TIME: Type <CR>
BASE PEAK INTENSITY: Type <CR>
USER PARAMETER # 1: Type <CR>
USER PARAMETER # 2: Type <CR>

RELATIVE RETENTION TIME: Type 1.000 <CR>
QUANTITATION MASS: Type 186 <CR>
STANDARD AMOUNT: Type 125 <CR>
UNITS: Type NG/CT <CR>
REFERENCE PEAK (NAME,NUM): Type RR,1 <CR>
RESPONSE FACTOR: Type <CR>

19.4.13 Disregard questions Retention Time and Base Peak Intensity because these questions are answered by the computer.

19.4.14 Disregard User Parameter #1 and #2 and Response Factor as these questions are not applicable at this point.

19.4.15 Calculate the Relative Retention Time by dividing the scan number of the target compound into the scan number of the standard (PFB).

19.4.16 Remember, the first entry of a quantitation library must be the ion of the standard compound against which quantitation is to be performed, so make the 186 ion of PFB the first entry.

19.4.17 Obtain the value for Standard Amount from the person who has loaded the cartridges.

NOTE: In library RR the Reference Peak and Number will always be answered RR,1 so that a library entry and an ion of a particular target compound can be correlated and quantitated against the standard ion.

19.4.18 After the questions have been answered, verify the correctness of all the information entered for the ion of a compound by inspecting the terminal display to be entered in library RR.

19.4.19 If no errors have been made in the entry of the information, simply strike <CR> to enter the EDLB program.

19.4.20 If there is more than one ion which needs to be entered for a given compound, type EDLB: I' <CR> again.

19.4.21 Answer all the questions in the same way except for the Quantitation Mass which must be changed to the other ion from the compound of interest.

19.4.22 If no change is necessary for a given answer, strike <CR>.

19.4.23 Should an error be discovered at the end of the list of questions, type N <CR> to enter EDLB.

19.4.24 Then type EDLB: I' <CR> to reenter the correct responses.

19.4.25 When all the ions desired for quantitation for a given compound have been entered into the library, return to the CHRO program by typing EDLB: E <CR> and LIBR: E <CR>.

19.4.26 Find the scan number for the next target compound and repeat the above steps for entry into the library until all the ions of all the compounds have been entered.

19.4.27 Obtain a list of all the library entries in library RR by typing >SET6 RR.LL <CR>.

19.4.28 Type >EDLL <CR>, followed by EDLL: 1,n where n is the total number of ions entered in library RR.

19.4.29 Type EDLL: B <CR> and a printout of library RR will appear at the Versatec printer.

19.4.30 Repeat all of the above steps for the compounds on the CPMX + RMR cartridges.

19.5 Correlation of Library Entry and Quantitation List

19.5.1 When both a Quantitation List and a library have been created, obtain calculated Response Factors by making a correlation between the ion of the compound and a library entry number.

19.5.2 Enter EDQL again by typing >EDQL file name <CR>.

19.5.3 Using library RR and a Quantitation List for an RMR cartridge type EDQL: C library RR <CR>.

NOTE: The computer then asks for the library name and the entry number that correlate to a compound ion. For example, when the 186 ion of PFB is listed type EDQL: RR,1 <CR> to correlate the first entry in library RR with the 186 ion of PFB.

19.5.4 Correlate all the ions to a library entry as they appear under EDQL.

NOTE: When all ions have been correlated, EDQL will display a summary table containing ions, scan number, peak area, library name, and entry number.

19.5.5 Verify that no errors have been made, then type EDQL: W <CR> to write the data back to the Quantitation List.

19.5.6 Exit from EDQL by typing EDQL: E <CR> and type > QUAN file name <CR>.

19.5.7 Under QUAN type QUAN: F2;H <CR> and a hardcopy of Response Factors for the various ions will be printed on the Versatec printer (see Figure 15).

19.5.8 Store these Response Factors in a Response List of the computer by typing QUAN: > <CR>.

19.5.9 Repeat these same steps for the CPMX + RMR quantitation list and the appropriate library.

19.6 Sample Quantitation

19.6.1 Write a procedure to allow quantitation of all desired ions of all target compounds to perform sample quantitation.

19.6.2 Create a library of all target compounds.

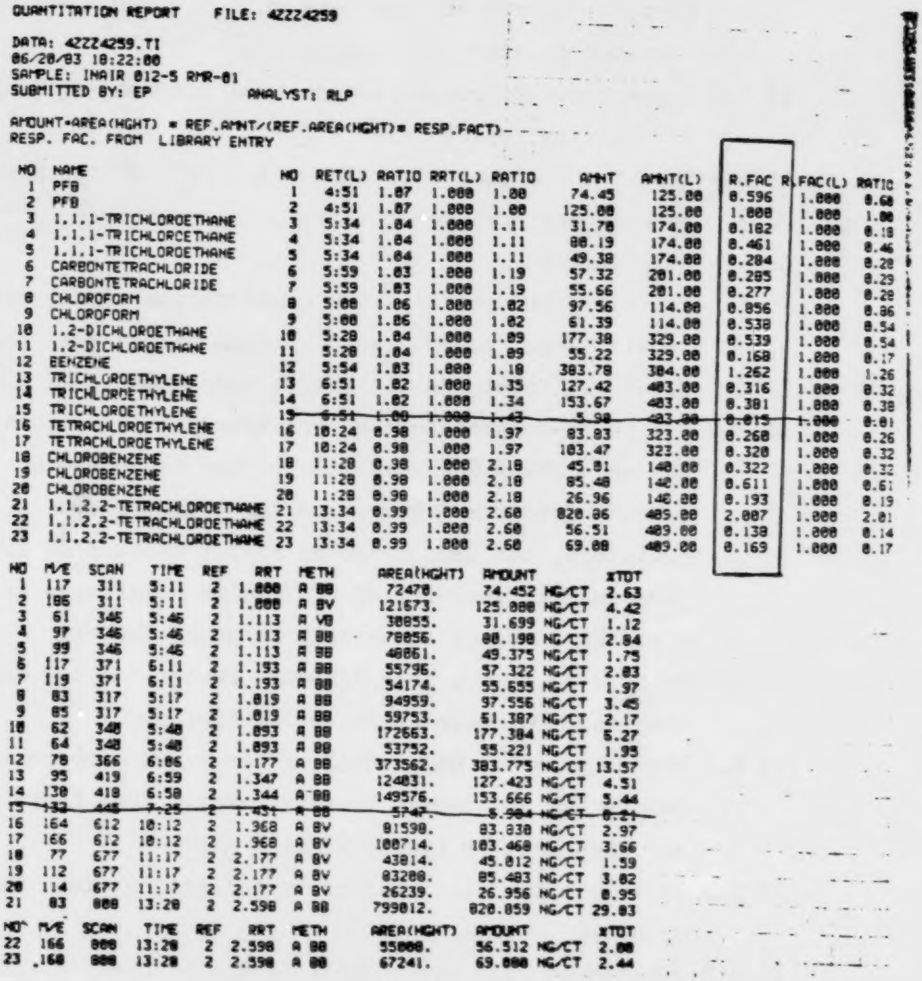
19.6.3 Use the same procedure as the Procedure and library for the RMR and CPMX + RMR compounds. When the compound ion is entered into the library and the computer asks a series of questions, the mean Response Factor calculated from the five initial analyses of CPMX + RMR cartridges is entered when the computer requests a Response Factor.

19.6.4 Be sure to list a mean Response Factor for all the compound ions in the sample quantitation library.

19.6.5 When a printout under QUAN of the final numbers is desired, delete the extraneous ions as before, type EDQL: W <CR>, and then exit by typing EDQL: E <CR>.

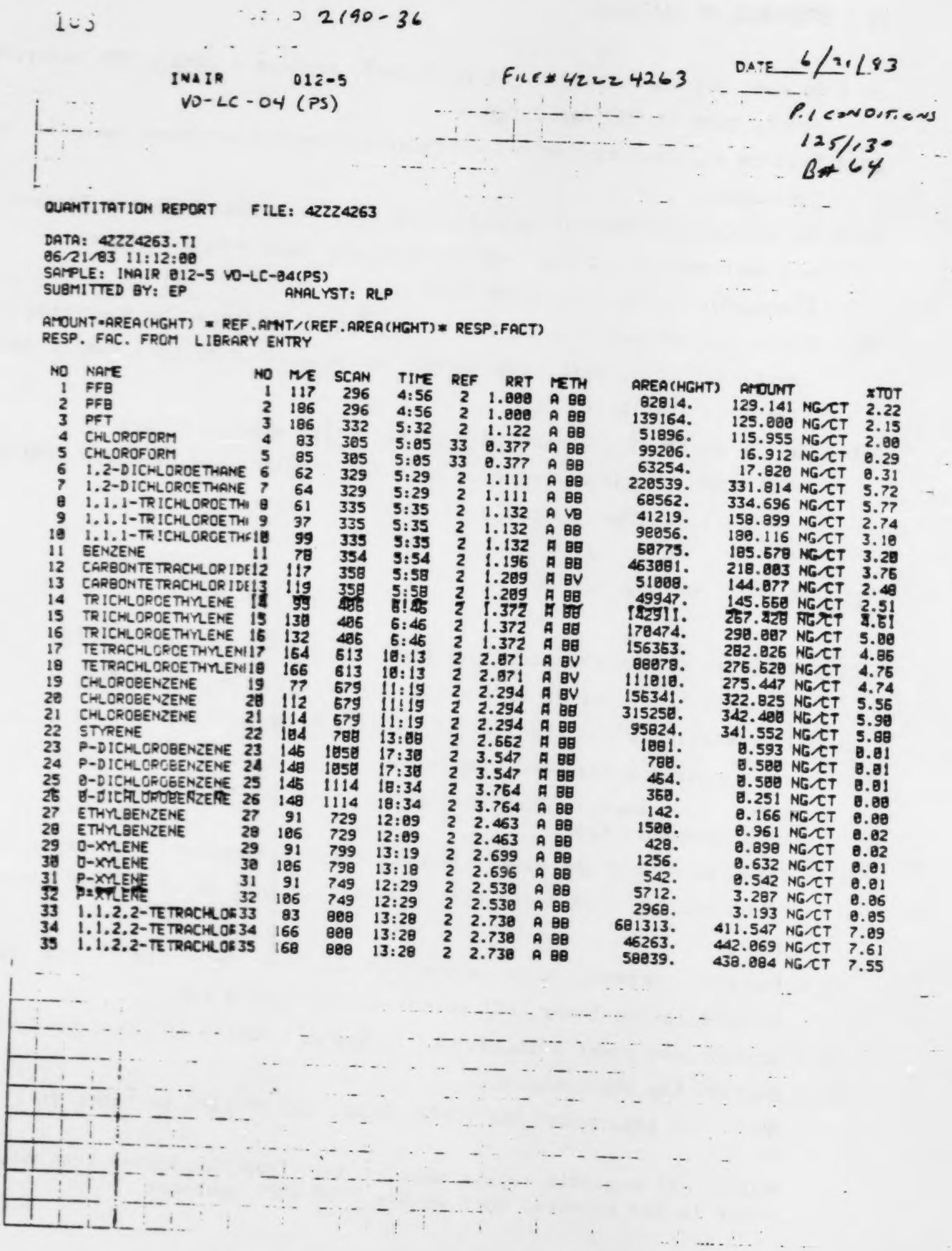
19.6.6 To obtain the final answer in ng/cart, go into the QUAN program.

19.6.7 Type >QUAN file name <CR> followed by QUAN: H <CR> (not QUAN: F2;H as before) (see Figure 16 for an example).



```
QUANTITATION REPORT    FILE: 4ZZ4259

DATA: 4ZZ4259.TI
06/20/83 10:22:00
SAMPLE: INAIR 012-5 RMR-01
SUBMITTED BY: EP              ANALYST: RLP

AMOUNT=AREA(HGHT) * REF.AMNT/(REF.AREA(HGHT)* RESP.FACT)
RESP. FAC. FROM  LIBRARY ENTRY

 NO  NAME
  1  PFB                             NO  RET(L) RATIO RRT(L) RATIO     AMNT   AMNT(L)   R.FAC R.FAC(L) RATIO
  2  PFB                              1   4:51  1.07  1.000  1.00     74.45   125.00   0.596   1.000   0.60
  3  1.1.1-TRICHLOROETHANE            2   4:51  1.07  1.000  1.00    125.00   125.00   1.000   1.000   1.00
  4  1.1.1-TRICHLORCETHANE            3   5:34  1.04  1.000  1.11     31.70   174.00   0.182   1.000   0.18
  5  1.1.1-TRICHLOROETHANE            4   5:34  1.04  1.000  1.11     80.19   174.00   0.461   1.000   0.46
  6  CARBONTETRACHLORIDE              5   5:34  1.04  1.000  1.11     49.38   174.00   0.284   1.000   0.28
  7  CARBONTETRACHLORIDE              6   5:59  1.03  1.000  1.19     57.32   201.00   0.285   1.000   0.29
  8  CHLOROFORM                       7   5:59  1.03  1.000  1.19     55.66   201.00   0.277   1.000   0.28
  9  CHLOROFORM                       8   5:00  1.06  1.000  1.02     97.56   114.00   0.856   1.000   0.86
 10  1.2-DICHLOROETHANE               9   5:00  1.06  1.000  1.02     61.39   114.00   0.538   1.000   0.54
 11  1.2-DICHLOROETHANE              10   5:20  1.04  1.000  1.09    177.38   329.00   0.539   1.000   0.54
 12  BENZENE                         11   5:20  1.04  1.000  1.09     55.22   329.00   0.168   1.000   0.17
 13  TRICHLOROETHYLENE               12   5:54  1.03  1.000  1.10    383.78   384.00   1.262   1.000   1.26
 14  TRICHLORCETHYLENE               13   6:51  1.02  1.000  1.35    127.42   403.00   0.316   1.000   0.32
 15  TRICHLOROETHYLENE               14   6:51  1.02  1.000  1.34    153.67   403.00   0.381   1.000   0.38
 16  TETRACHLOROETHYLENE           ~~15   6:51  1.00  1.000  1.43      5.90   403.00   0.015   1.000   0.01~~
 17  TETRACHLOROETHYLENE             16  10:24  0.98  1.000  1.97     83.83   323.00   0.260   1.000   0.26
 18  CHLOROBENZENE                   17  10:24  0.98  1.000  1.97    103.47   323.00   0.320   1.000   0.32
 19  CHLOROBENZENE                   18  11:28  0.98  1.000  2.18     45.81   140.00   0.322   1.000   0.32
 20  CHLOROBENZENE                   19  11:28  0.98  1.000  2.18     85.48   142.00   0.611   1.000   0.61
 21  1.1.2.2-TETRACHLOROETHANE       20  11:28  0.98  1.000  2.18     26.96   146.00   0.193   1.000   0.19
 22  1.1.2.2-TETRACHLOROETHANE       21  13:34  0.99  1.000  2.60    820.86   405.00   2.007   1.000   2.01
 23  1.1.2.2-TETRACHLOROETHANE       22  13:34  0.99  1.000  2.60     56.51   409.00   0.138   1.000   0.14
                                     23  13:34  0.99  1.000  2.60     69.08   409.00   0.169   1.000   0.17

 NO  M/E  SCAN   TIME  REF   RRT  METH    AREA(HGHT)  AMOUNT           %TOT
  1  117   311   5:11   2  1.000  A BB       72470.     74.452 NG/CT   2.63
  2  186   311   5:11   2  1.000  A BV      121673.    125.000 NG/CT   4.42
  3   61   346   5:46   2  1.113  A VB       30855.     31.699 NG/CT   1.12
  4   97   346   5:46   2  1.113  A BB       78056.     80.190 NG/CT   2.84
  5   99   346   5:46   2  1.113  A BB       48061.     49.375 NG/CT   1.75
  6  117   371   6:11   2  1.193  A BB       55796.     57.322 NG/CT   2.03
  7  119   371   6:11   2  1.193  A BB       54174.     55.655 NG/CT   1.97
  8   83   317   5:17   2  1.019  A BB       94959.     97.556 NG/CT   3.45
  9   85   317   5:17   2  1.019  A BB       59753.     61.387 NG/CT   2.17
 10   62   348   5:40   2  1.093  A BB      172663.    177.384 NG/CT   6.27
 11   64   348   5:40   2  1.093  A BB       53752.     55.221 NG/CT   1.95
 12   78   366   6:06   2  1.177  A BB      373562.    383.775 NG/CT  13.57
 13   95   419   6:59   2  1.347  A BB      124031.    127.423 NG/CT   4.51
 14  130   418   6:58   2  1.344  A BB      149576.    153.666 NG/CT   5.44
~~15  132   445   7:25   2  1.431  A BB        5747.      5.904 NG/CT   0.21~~
 16  164   612  10:12   2  1.368  A BV       81590.     83.830 NG/CT   2.97
 17  166   612  10:12   2  1.968  A BV      100714.    103.460 NG/CT   3.66
 18   77   677  11:17   2  2.177  A BV       43814.     45.012 NG/CT   1.59
 19  112   677  11:17   2  2.177  A BV       83200.     85.483 NG/CT   3.02
 20  114   677  11:17   2  2.177  A BV       26239.     26.956 NG/CT   0.95
 21   83   800  13:20   2  2.598  A BB      799012.    820.859 NG/CT  29.03

 NO  M/E  SCAN   TIME  REF   RRT  METH    AREA(HGHT)  AMOUNT           %TOT
 22  166   800  13:20   2  2.598  A BB       55000.     56.512 NG/CT   2.00
 23  168   800  13:20   2  2.598  A BB       67241.     69.000 NG/CT   2.44
```

Figure 15. Quantitation report for a RMR run showing response factors.



[illegible] 2190-36

INAIR 012-5 VO-LC-04 (PS)

FILE# 42ZZ4263

DATE 6/21/83

P.I CONDITIONS
125/130
B# 64

```
QUANTITATION REPORT    FILE: 42ZZ4263

DATA: 42ZZ4263.TI
06/21/83 11:12:00
SAMPLE: INAIR 012-5 VO-LC-04(PS)
SUBMITTED BY: EP              ANALYST: RLP

AMOUNT=AREA(HGHT) * REF.AMNT/(REF.AREA(HGHT)* RESP.FACT)
RESP. FAC. FROM  LIBRARY ENTRY

NO  NAME                NO  M/E  SCAN   TIME  REF   RRT  METH   AREA(HGHT)  AMOUNT          %TOT
 1  PFB                  1  117   296   4:56   2  1.000  A BB      82814.   129.141 NG/CT  2.22
 2  PFB                  2  186   296   4:56   2  1.000  A BB     139164.   125.000 NG/CT  2.15
 3  PFT                  3  186   332   5:32   2  1.122  A BB      51896.   115.955 NG/CT  2.00
 4  CHLOROFORM           4   83   305   5:05  33  0.377  A BB      99206.    16.912 NG/CT  0.29
 5  CHLOROFORM           5   85   305   5:05  33  0.377  A BB      63254.    17.820 NG/CT  0.31
 6  1.2-DICHLOROETHANE   6   62   329   5:29   2  1.111  A BB     220539.   331.814 NG/CT  5.72
 7  1.2-DICHLOROETHANE   7   64   329   5:29   2  1.111  A BB      68562.   334.696 NG/CT  5.77
 8  1.1.1-TRICHLOROETHA  8   61   335   5:35   2  1.132  A VB      41219.   158.899 NG/CT  2.74
 9  1.1.1-TRICHLOROETHA  9   97   335   5:35   2  1.132  A BB      98056.   180.116 NG/CT  3.10
10  1.1.1-TRICHLOROETHA10   99   335   5:35   2  1.132  A BB      60775.   185.670 NG/CT  3.20
11  BENZENE             11   78   354   5:54   2  1.196  A BB     463081.   218.003 NG/CT  3.76
12  CARBONTETRACHLORIDE12  117   358   5:58   2  1.209  A BV      51008.   144.077 NG/CT  2.48
13  CARBONTETRACHLORIDE13  119   358   5:58   2  1.209  A BB      49947.   145.560 NG/CT  2.51
14  TRICHLOROETHYLENE   14   95   406   6:46   2  1.372  A BB     142911.   267.428 NG/CT  4.61
15  TRICHLOROETHYLENE   15  130   406   6:46   2  1.372  A BB     170474.   290.807 NG/CT  5.00
16  TRICHLOROETHYLENE   16  132   406   6:46   2  1.372  A BB     156363.   282.026 NG/CT  4.86
17  TETRACHLOROETHYLENE17  164   613  10:13   2  2.071  A BV      88078.   276.520 NG/CT  4.76
18  TETRACHLOROETHYLENE18  166   613  10:13   2  2.071  A BV     111010.   275.447 NG/CT  4.74
19  CHLOROBENZENE       19   77   679  11:19   2  2.294  A BV     156341.   322.825 NG/CT  5.56
20  CHLOROBENZENE       20  112   679  11:19   2  2.294  A BB     315250.   342.400 NG/CT  5.90
21  CHLOROBENZENE       21  114   679  11:19   2  2.294  A BB      95824.   341.552 NG/CT  5.88
22  STYRENE             22  104   780  13:00   2  2.662  A BB       1001.     0.593 NG/CT  0.01
23  P-DICHLOROBENZENE   23  146  1050  17:30   2  3.547  A BB        780.     0.500 NG/CT  0.01
24  P-DICHLOROBENZENE   24  148  1050  17:30   2  3.547  A BB        464.     0.500 NG/CT  0.01
25  O-DICHLOROBENZENE   25  146  1114  18:34   2  3.764  A BB        360.     0.251 NG/CT  0.00
26  O-DICHLOROBENZENE   26  148  1114  18:34   2  3.764  A BB        142.     0.166 NG/CT  0.00
27  ETHYLBENZENE        27   91   729  12:09   2  2.463  A BB       1500.     0.961 NG/CT  0.02
28  ETHYLBENZENE        28  106   729  12:09   2  2.463  A BB        428.     0.898 NG/CT  0.02
29  O-XYLENE            29   91   799  13:19   2  2.699  A BB       1256.     0.632 NG/CT  0.01
30  O-XYLENE            30  106   798  13:18   2  2.696  A BB        542.     0.542 NG/CT  0.01
31  P-XYLENE            31   91   749  12:29   2  2.530  A BB       5712.     3.287 NG/CT  0.06
32  P-XYLENE            32  106   749  12:29   2  2.530  A BB       2968.     3.193 NG/CT  0.05
33  1.1.2.2-TETRACHLOR33   83   808  13:28   2  2.730  A BB     681313.   411.547 NG/CT  7.09
34  1.1.2.2-TETRACHLOR34  166   808  13:28   2  2.730  A BB      46263.   442.069 NG/CT  7.61
35  1.1.2.2-TETRACHLOR35  168   808  13:28   2  2.730  A BB      58039.   438.084 NG/CT  7.55
```

Figure 16. Completed sample quantitation report.

20.0 SEQUENCE OF SAMPLES

20.1 On each day that analysis is performed, analyze a CPMX + RMR cartridge first, then an RMR cartridge.

20.2 Perform all Quality Control calculations before any more analysis is initiated.

20.3 If the Quality Control calculations, PFT tune, and Response Factors all meet specifications, analyze a blank Tenax sample (either a laboratory blank or a field blank).

20.4 Following the analysis of the blank sample, analyze the remaining cartridges in no particular order, but analyze a control sample daily if one is available.

20.5 At the conclusion of each day of analysis, analyze either a CPMX + RMR or RMR cartridge, so that a complete set of three CPMX + RMR and RMR cartridges is analyzed every two days.

21.0 STORAGE OF DATA

21.1 Transfer all data acquired on the Finnigan 4021 to nine-track magnetic tapes for archiving and possible further reference.

21.2 If possible, avoid transfer of data to magnetic tape until all processing has been completed.

NOTE: Two complete disk images can be stored on a single 1200' magnetic tape.

21.3 Take two data disks where all data are processed and put one in unit #0 of the top disk drive and the other in unit #2 of the bottom disk drive.

21.4 Install a magnetic tape on the tape drive by referring to the diagram located in the lower left corner of the tape drive.

21.5 Depress the power switches at the top and bottom of the tape drive.

21.6 Depress the LOAD switch.

NOTE: At this point the POWER, LOAD, and ON LINE switches will be lit.

NOTE: All magnetic taping must be done from the master terminal which is the terminal used by the FINN 4021 operator.

21.7 On the master terminal type > ID <CR> and a + will appear on the screen.

21.8 Type + DTDW <CR> followed by H (no CR).

NOTE: A list of choices describing the capabilities of this program will appear.

21.9 Transfer one disk image at a time to tape.

NOTE: No carriage returns are necessary in the DTDW program.

21.10 Tell the computer initially what disk unit is to be copied by typing + U followed by the disk number to be taped.

21.11 Type + D and the first disk will be copied to the magnetic tape.

NOTE: This is considered the first file on the tape.

21.12 For the second disk type + U and disk number also.

21.13 Type + D again.

21.14 When both disk images are copied depress RESET and REWIND buttons on the tape drive to rewind the tape back to the LOAD position.

21.15 Depress the ON LINE switch.

21.16 Verify the disk images to ensure no error has been made in copying to the magnetic tape.

21.17 Type + U and the disk number copied on the first file.

21.18 Type + V.

NOTE: The same procedure is followed for the second disk image.

21.19 When both images are verified type + B to bootstrap the computer back to the MSDS operating system.

21.20 Remove the magnetic tape from the tape deck.

21.21 Replace the tape in the tape seal.

21.22 Label the tape with the next number for the sequence for Finnigan 4021 tapes, either XYZ,1 for the first tape file or XYZ,2 for the second tape file.

21.23 Make the appropriate entry in the tape log.

21.24 Make the appropriate entry in the instrument log so that a data file is readily correlated with a magnetic tape and one of the two files on that tape.

22.0 QUALITY CONTROL

Quality Control for the Finnigan 4021 requires calculations on peaks from the CPMX + RMR cartridge, the % RA of major ions of PFT, and response factors of the target compounds in the CPMX + RMR and RMR samples. It is extremely important these criteria be met before running any samples to ensure quality data.

22.1 Peak Asymmetry Calculations

22.1.1 Utilize a column performance Tenax cartridge to assure that the GM/MS/DS is operating properly.

22.1.2 Check the instrument tune stability as well as capillary column performance.

22.1.3 Calculate a peak asymmetry to measure peak tailing for the compounds acetophenone, 5-nonanone, and 1-octanol.

NOTE: The formula is % PAF = F/B x 100 where B equals the width of the back half of the peak 10% above the baseline and F equals the width of the front half of the peak 10% above the baseline (see Figure 17). The specifications acceptable for these compounds are as follows:

Acetophenone	< 300
5-Nonanone	< 160
1-Octanol	< 250

22.2 Separation Number

22.2.1 Calculate the separation number for octane and decane to measure the separation efficiency of the capillary column.

NOTE: A value > 40 is acceptable. The formula is as follows:

$$SN = \left(\frac{D}{W_1+W_2}\right) - 1$$

where D equals the distance between the two peaks on the RIC whereas W_1 and W_2 equal the width of the peak at 1/2 height (see Figure 18).

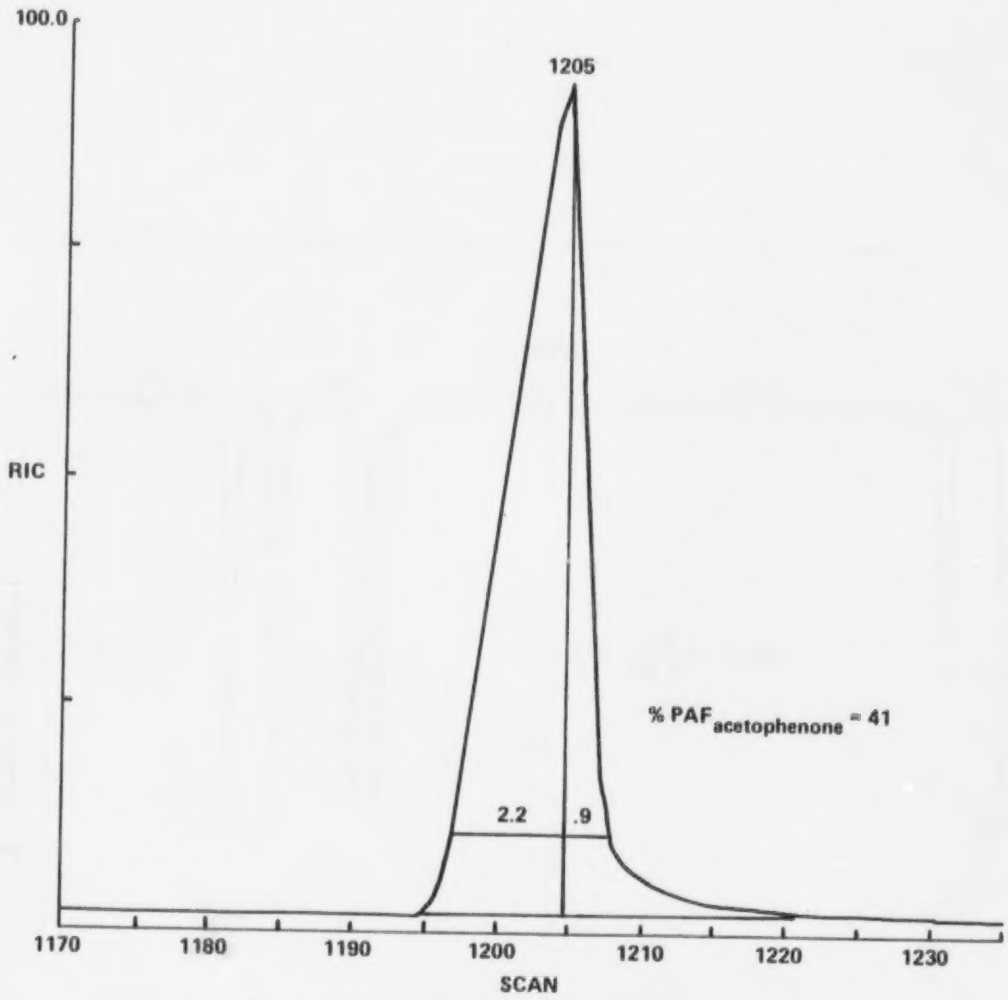


Figure 17. Peak asymmetry for acetophenone.

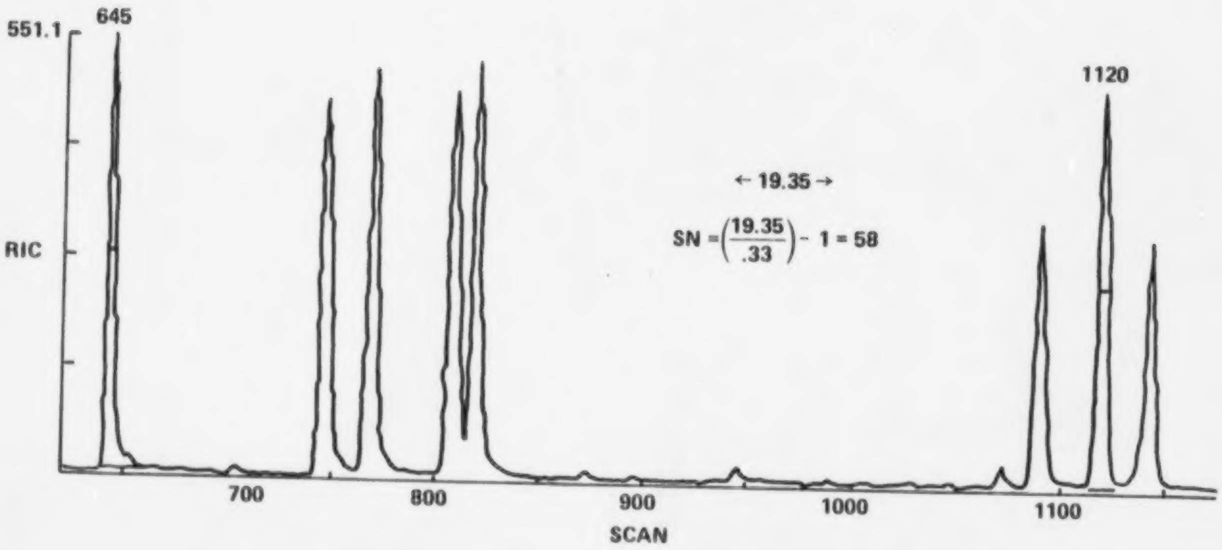


Figure 18. Separation number between octane and decane.

22.3 Resolution

22.3.1 Calculate resolution by measuring ethylbenzene and p-xylene peaks in the RIC.

NOTE: The formula is as follows:

$$R = \frac{2D}{W_1+W_2}$$

where D equals the distance between the two peaks and W_1, W_2 equal the width of each peak on the baseline. This value must be > 1.0 (see Figure 19).

22.4 Acid/Base Ratios

22.4.1 To obtain the last column performance parameters, calculate an acid/base ratio between RIC areas of 2,6-dimethylaniline (DMA) and 2,6-dimethylphenol (DMP) versus RIC area of acetophenone.

NOTE: The formulas for acidity and basicity are as follows:

$$\text{Acidity} = \frac{\text{RIC area DMA}}{\text{RIC area acetophenone}}$$

$$\text{Basicity} = \frac{\text{RIC area DMP}}{\text{RIC area acetophenone}}$$

Acceptable values for these parameters range from 0.7 to 1.3.

22.5 Mass Spectrum of PFT

22.5.1 Obtain a listing of PFT as well as a plotted mass spectrum showing the % RA of the major ions.

22.5.2 Check the tune stability by comparing % RA to the historical % RA of the Finnigan 4021.

22.6 Computer Procedure for Column Performance Calculations

All data needed for column performance calculations can be obtained through the CHRO program. A procedure named CPMXI has been written to automatically hardcopy all RIC chromatograms needed. To utilize this procedure simply type > CPMXI <CR> after the CPMX + RMR sample has been run (see Figure 20) or generate appropriate hardcopy by individual instructions.

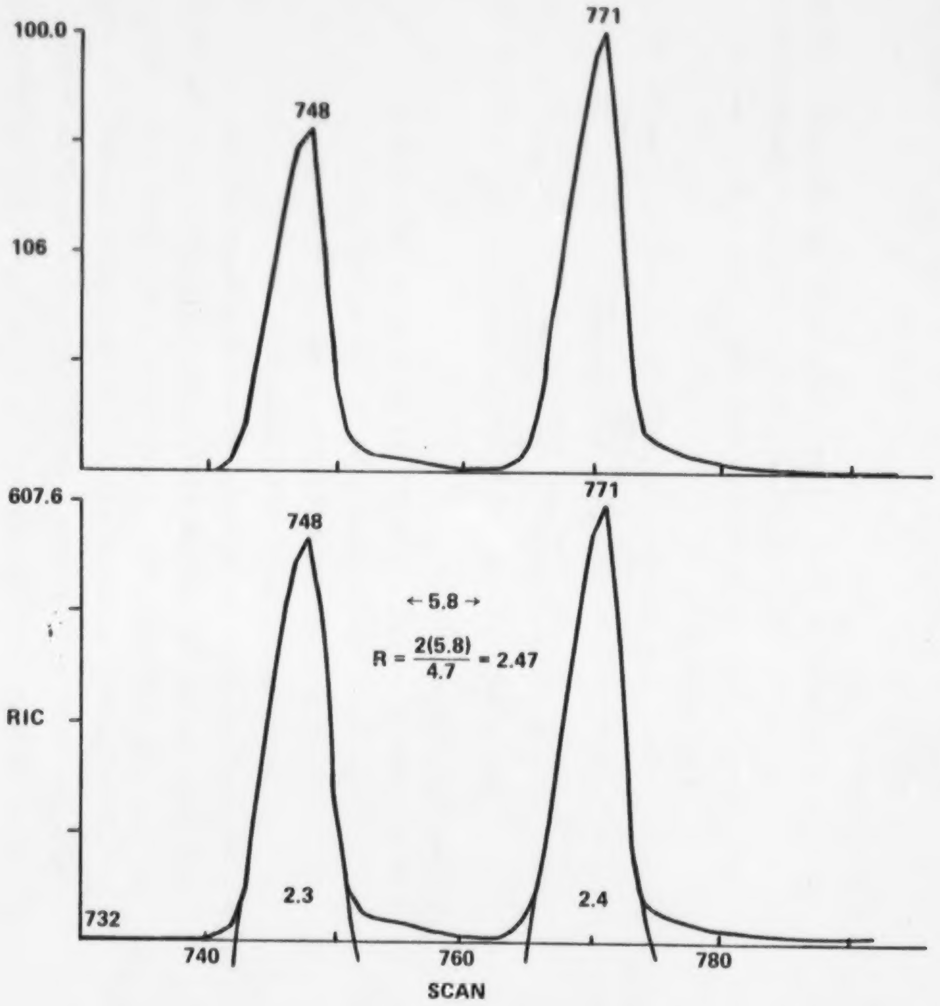


Figure 19. Resolution between ethylbenzene and p-xylene.

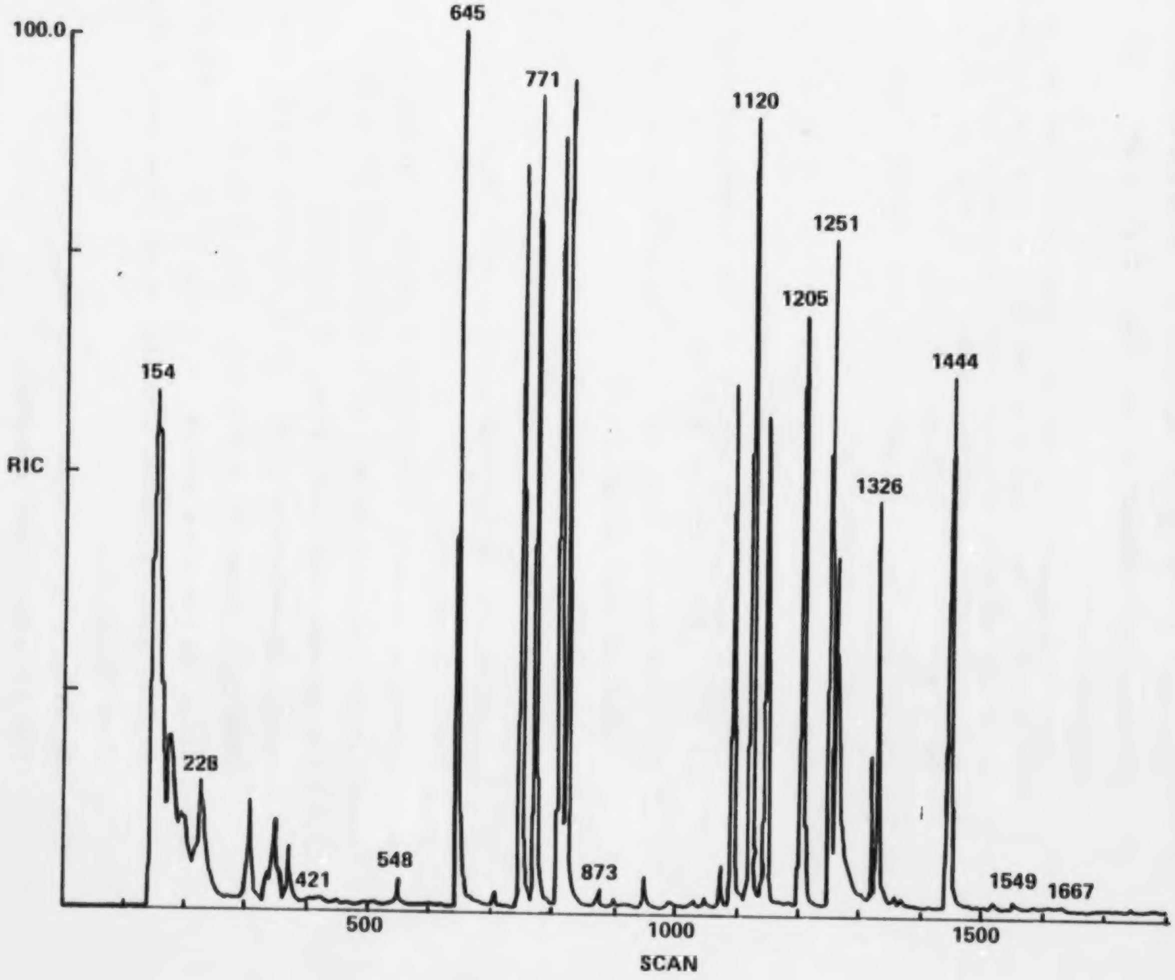


Figure 20. Typical CPMX and RMR run.

22.7 Response Factors

22.7.1 Once the mean response factors have been calculated for the five initial CPMX + RMR and RMR runs, make up a table consisting of the range of numbers within which the response factor must fall for each day of sample analysis.

22.7.2 Determine this range by multiplying the standard deviation by three and subtracting and adding this value to the mean response factor.

NOTE: For example, if the mean response factor of the 97 ion of 1,1,1-Trichloroethane is 1.00 and the standard deviation is 0.20, then the acceptable range would be 0.40-1.60. This range determines if a response factor for a given ion is "in" or "out" of control.

22.7.3 If only one ion of a compound is "in" control, then classify the entire compound "in" control.

22.7.4 Classify a compound as "out" of control only if all ions of a compound are "out" of control.

NOTE: This criterion applies to the target compounds of both the CPMX + RMR and RMR runs.

22.7.5 If the number of compounds "out" of control exceeds seven, analyze another standard cartridge.

22.7.6 If the number of compounds "out" of control is still not acceptable, check the instrument tune, test for an air leak, or establish whether some other problem is present and should be checked.

22.7.7 Tabulate the CPMX + RMR and RMR response factors for each day.

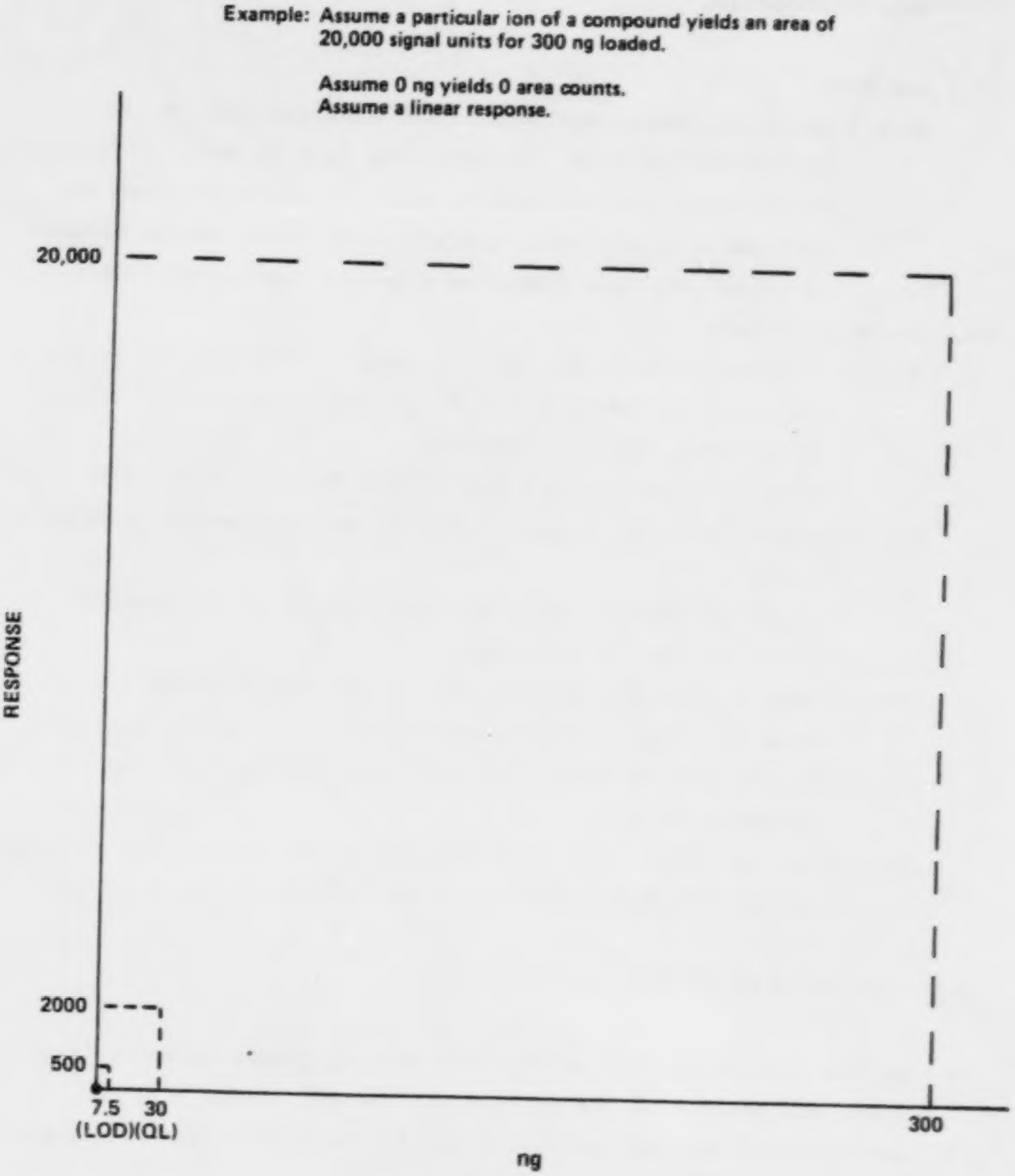
22.8 Estimation of Limits of Detection and Quantifiable Limits

22.8.1 Use an arbitrary noise level of 500 area counts to calculate limits of detection (LOD). Obtain a quantifiable limit of 2000 area counts by multiplying the LOD by four.

22.8.2 Using the raw area counts for a representative CPMX + RMR or RMR, plot ng loaded (X-axis) versus area counts (Y-axis) (see Figure 21).

22.8.4 By using a linear regression on the TI-55 calculator compute LOD for each target compound.

NOTE: The quantifiable limit is defined as four times the LOD.



Example: Assume a particular ion of a compound yields an area of 20,000 signal units for 300 ng loaded.

Assume 0 ng yields 0 area counts.
Assume a linear response.

Figure 21. LOD plot.

23.0 DATA BOOKKEEPTING

23.1 Log Book

23.1.1 List each sample analyzed on the Finnigan 4021 in the instrument log book, including the date of data acquisition, project number, mode of sample introduction, instrument conditions, type of column used, amount or type of injection, file name, and magnetic tape identification.

23.2 Project Notebook

23.2.1 List each CPMX + RMR, RMR, or sample cartridge run in the project notebook with a file name, date of acquisition, sample name, and GC conditions.

23.2.2 Write information from the Custody Sheet such as batch number of Tenax, temperature, volume, and amount of PFB/PFT loaded.

23.2.3 Leave the Custody sheet for each sample in the notebook until the end of the study.

23.2.4 Tape a chromatogram from each run in the notebook along with the final quantitation report of calculated ng/cart.

23.2.5 In the case of CPMX + RMR and RMR, keep the printout of response factors.

23.2.6 For the CPMX + RMR run, keep the column performance calculations of the RIC hardcopies in the project notebook.

24.0 CONCLUSION OF SAMPLE QUANTITATION

24.1 At the conclusion of a study, duplicate all sample quantitation lists and match with the appropriate Custody Sheet.

24.2 Double-check against the instrument log book to assure all samples have been quantitated and copied.

24.3 Relinquish data and Custody Sheets to the appropriate individual.

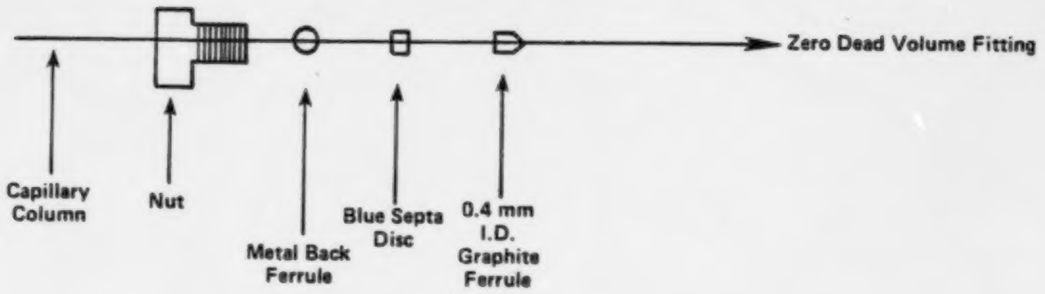

Zero Dead Volume Fitting
Capillary Column
Nut
Metal Back Ferrule
Blue Septa Disc
0.4 mm I.D. Graphite Ferrule

Figure 5. Front end of GC column.

RTI/ACS-SOP-533-002
April 1984
Revision 0

Standard Operating Procedure for the Analysis of Volatile Organics Collected on Tenax Using the Finnigan 3300 GC/MS/DS

Author:

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Approved:

Joan T. Bursey 4/19/84
Laboratory Manager, RTI/ACS Date

RWHandy 4/18/84
QA Officer, RTI/ACS Date

E Pellizzari 4/18/84
Vice-President, RTI/ACS Date

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Standard Operating Procedure for the Analysis of Volatile Organics Collected on Tenax Using the Finnigan 3300 GC/MS/DS

RTI/ACS-SOP-533-002

1.0 SCOPE AND APPLICATION

This standard operating procedure is applicable to the qualitative and quantitative analysis of volatile organic compounds collected on Tenax GC®. The method is not applicable as herein described to the analysis of permanent gases present in the atmosphere. In general, recovery from Tenax for compounds less volatile than ten-twelve carbon hydrocarbons is less than quantitative, so the method is not applicable to the quantitative analysis of the less volatile compounds, although many compounds of low volatility can be observed by thermal desorption from Tenax and analyzed qualitatively. Thermal desorption of Tenax followed by cryofocussing of the organic vapors, with subsequent capillary gas chromatography/mass spectrometry/data system analysis has been applied to adsorbed volatile organic compounds collected from exterior and interior air, personal air, air collected in the workplace, breath, and volatile organics transferred to Tenax from other adsorptive media. The method is limited by adsorption of water on the Tenax: if too much water is adsorbed, the cryofocussing trap will freeze and quantitative analysis will be impossible. The basic method is adaptable to any gas chromatograph/mass spectrometer/computer system upon construction of a suitable thermal desorption unit. A certain amount of flexibility in the analytical method from instrument to instrument is tolerable in order to optimize the operating parameters of any given instrument. Data handling procedures may also follow a wide range (from completely computerized to entirely manual) and still produce data within the criteria for acceptability. The following Standard Operating Procedure is related to optimal operating procedures for a particular instrument.

2.0 SUMMARY OF THE PROCEDURE

Tenax GC® is a porous p-phenylene oxide polymer which preferentially adsorbs volatile organic compounds. The affinity of this polymer for water is low, and thus it is possible to sample many liters of air (often 20-30 or more) without adsorbing so much water that application of analytical procedures becomes prohibitive. The affinity of this polymer for organic vapors makes its cleaning and preparation for field sampling crucial to the success of the ultimate analysis. Once the sample cartridge has been exposed in the field, extremely careful handling is required in order to avoid contamination. The sample cartridge, shipped back from the field, is loaded with a standard (or standards) to permit quantitative analysis. The cartridge is then submitted for analysis by capillary gas chromatograph/mass spectrometer/data system. The organic vapors are removed from the Tenax by heating the sample cartridge under a flow of helium. The desorbed vapors are collected in a trap which is cooled to liquid nitrogen temperatures. This step is essential to focus the organic compounds and allow their application to the head of the capillary column in a discrete band. A cartridge heater raises the temperature of the trap from liquid nitrogen temperatures (-198°C) to 250°C in approximately 2.5 minutes, vaporizing all of the entrapped organics very quickly. The vaporized organics are transferred to the head of the capillary column which is at or near room temperature, the scan of the mass spectrometer is initiated, and the analytical procedure is begun. The column is programmed to a temperature to allow the elution of all of the organic compounds while the mass spectrometer is scanning. Data are recorded by the computer for subsequent processing. Quantitation is performed by the method of Relative Response Factors, where the proportionate system responses for analyte and standard are determined prior to the analysis of the sample and this relative system response is used to determine the quantity of compound present on the sample cartridge. The quantitative analysis is performed by a combination of manual and computerized procedures: the computer is instructed to seek characteristic ions in a previously determined retention window. At this point the operator intervenes to determine if the compound of interest has been located correctly. If the compound identification is correct, the computer then performs the quantitative calculation using the method of Relative Response Factors. Data are reported as ng/cartridge, and can be subsequently converted to whatever units are desired.

Quality control procedures are followed in order to determine that the column is performing within acceptable limits, the mass spectrometer is tuned correctly and performing acceptably, and chromatography criteria are being met. A chromatogram (actually reconstructed ion chromatogram) is obtained for each analysis and entered into the laboratory project notebook. The quantitation report from the computer is also entered into the project notebook. Standard Chain-of-Custody procedures are followed for every sample analyzed. The deliverables include a copy of the computer-generated quantitation report and the Chain-of-Custody form. Also, an entry is made into the appropriate instrument log for every sample analyzed.

3.0 PREPARATION OF GOLD-PLATED NICKEL TUBING

Gold-plated nickel tubing is required for an inert transfer line between the desorption unit and the capillary column. This tubing must be prepared, as it is not commercially available.

3.1 Materials and Equipment

3.1.1 1/16" o.d. nickel tubing (15") (Supelco)
3.1.2 Aspirator
3.1.3 Large beaker (Corning)
3.1.4 Heater (Fisher)
3.1.5 Teflon tubing (Penntube)
3.1.6 Atomex (AuCN) (Englehard)
3.1.7 Distilled water (Research Triangle Institute)
3.1.8 Acetone (Fisher)
3.1.9 20 mL 1N HCl (Fisher)
3.1.10 Thermometer (Supelco)
3.1.11 Solution of 10% OV-17 in acetone (Supelco)

3.2 Procedure

3.2.1 Fill large beaker with water and submerge the coiled nickel tubing (see Figure 1).

3.2.2 Using the aspirator, pull 20-40 mL acetone through the tubing.

NOTE: This rinse with acetone will remove trace organic compounds from the tubing.

3.2.3 Heat water bath to 70°C.

3.2.4 Pull 20 mL 1N HCl through the nickel tubing.

3.2.5 Rinse the acid from the tubing by pulling through 50 mL distilled water.

3.2.6 Draw 20 mL of Atomex through the tubing at a rate of 1 mL/min.

NOTE: This amount of Atomex is only an estimate; more or less may be required to complete the plating process. The amount of plating can be checked by inspecting the end of the tubing immersed in the Atomex which was last reached by the Atomex.

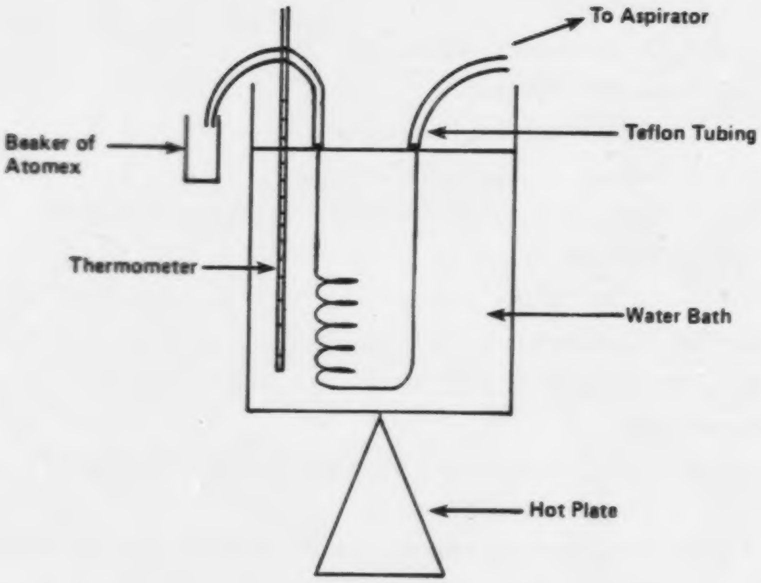

To Aspirator
Teflon Tubing
Beaker of Atomex
Thermometer
Water Bath
Hot Plate

Figure 1. Diagram for gold plating nickel tubing.

3.2.7 Rinse the tubing with distilled water, HCl, and distilled water again.

WARNING

Be extremely careful not to allow any mixing of Atomex (AuCN) with any of the HCl rinses in the aspirator flask. If Atomex and HCl are allowed to mix, toxic HCN is formed.

3.2.8 Rinse with acetone.

3.2.9 Pull OV-17 solution through tubing at a rate of approximately 1 mL/min.

4.0 INSPECTION AND PREPARATION OF THERMAL DESORPTION UNIT

4.1 Materials

4.1.1 Six-port high temperature valve (Valco)

4.1.2 25 mm Teflon cap, 5 mm thick (fabricated at Research Triangle Institute)

4.1.3 Nickel trap with 1/16" o.d. nickel tubing (fabricated at Research Triangle Institute)

4.1.4 Aluminum block to accept cartridge (fabricated at the Research Triangle Institute)

4.1.5 Block heaters (2) (Hotwatt)

4.1.6 Thermocouples (6) (fabricated at the Research Triangle Institute)

4.1.7 1/16" male nuts (6) (Valco)

4.1.8 1/16" ferrules (6) (Valco)

4.1.9 Desorption chamber and cap handle (fabricated at the Research Triangle Institute)

4.1.10 Aluminum foil (grocery store)

4.1.11 Cotter pin (hardware store)

4.1.12 Insulation

4.1.13 Heater for transfer line (fabricated at the Research Triangle Institute)

4.1.14 Valve stem and handle (Valco)

4.1.15 Front and back cover plates (fabricated at the Research Triangle Institute)

4.1.16 Screws (hardware store)

4.1.17 Melting point capillary tubes (Supelco)

4.1.18 1/16" o.d. stainless steel tubing (8") (Supelco)

4.1.19 1/16" o.d. gold-plated nickel tubing (15") (tubing from Supelco, gold-plated at the Research Triangle Institute)

4.1.20 Allen wrench (Sears)

4.1.21 Flat-bladed screwdriver (Sears)

4.1.22 7/16" open-end wrench (Sears)

4.1.23 Temperature controller for thermal desorption unit (fabricated at the Research Triangle Institute)

4.1.24 Metal mounting bracket for thermal desorption unit, to mount unit to the gas chromatograph (fabricated at the Research Triangle Institute)

4.1.25 1/8" copper tubing (several feet) (Dillon Supply)

4.1.26 Fine metering valve (Hoke)

4.1.27 Toggle valve (Raleigh Valve and Fitting)

4.1.28 Helium (2 tanks) (National Welders)

4.1.29 Helium regulators (2) (Matheson)

4.1.30 T-bar (Swagelok)

4.1.31 1/4"-1/8" female pipe fitting (2) (Swagelok)

4.1.32 Bubble flow meter (Supelco)

4.1.33 Metal column tags (Supelco)

4.1.34 Snoop (Nupro)

4.2 Disassembly of the Thermal Desorption Unit to Permit Inspection

4.2.1 Remove the back cover plate by extracting four screws with a flat-bladed screwdriver.

NOTE: With the cover plate removed, the inner fiberglass insulation can be removed. Use gloves when removing the insulation. Particles of fiberglass on the skin will be irritating.

4.2.2 Extract the insulation, setting it carefully aside for later reinsertion, to make the back half of the aluminum block visible.

4.2.3 Dismount the desorption unit from above the oven of the gas chromatograph by removing two screws connected to its base with a flat-bladed screwdriver.

4.2.4 Lay the dismounted unit face down on a table, being very careful not to displace any thermocouple or heater wires.

4.2.5 Using a 5/32" Allen wrench, remove the four bolts from the aluminum block to allow the six-port high temperature valve and all of its associated connections (see Figure 2) to be visible.

4.2.6 Now that the unit has been disassembled to this point, initiate the inspection.

4.3 Inspection of the Thermal Desorption Unit

4.3.1 Verify that all fittings which connect to the six-port valve are tight, using a wrench to tighten them if necessary.

4.3.2 Verify that a heater and two thermocouples are wrapped securely with aluminum foil around the gold-plated transfer line.

4.3.3 Check that the thermocouples are encased in a short length of glass capillary tubing to prevent shorting out against metal surfaces and loss of a temperature reading.

4.3.4 Verify that two thermocouples encased in glass melting point capillaries are located on the side of the front piece of the aluminum block.

NOTE: The unit will not give temperature readouts which are crucial to the maintenance of proper operating temperatures if the thermocouples are not securely encased in glass or if the glass is cracked. These glass tubes must cover the length of exposed wire of the thermocouple.

4.3.5 Check that the nickel lines connected to the valve and extending to the trap are covered with aluminum foil until the lines start to coil at the trap.

4.3.6 Verify that a heater is located at the top of the trap.

4.3.7 Check to be certain that the "trap in" line actually goes down to the bottom of the trap, and that the "trap out" line coils back up.

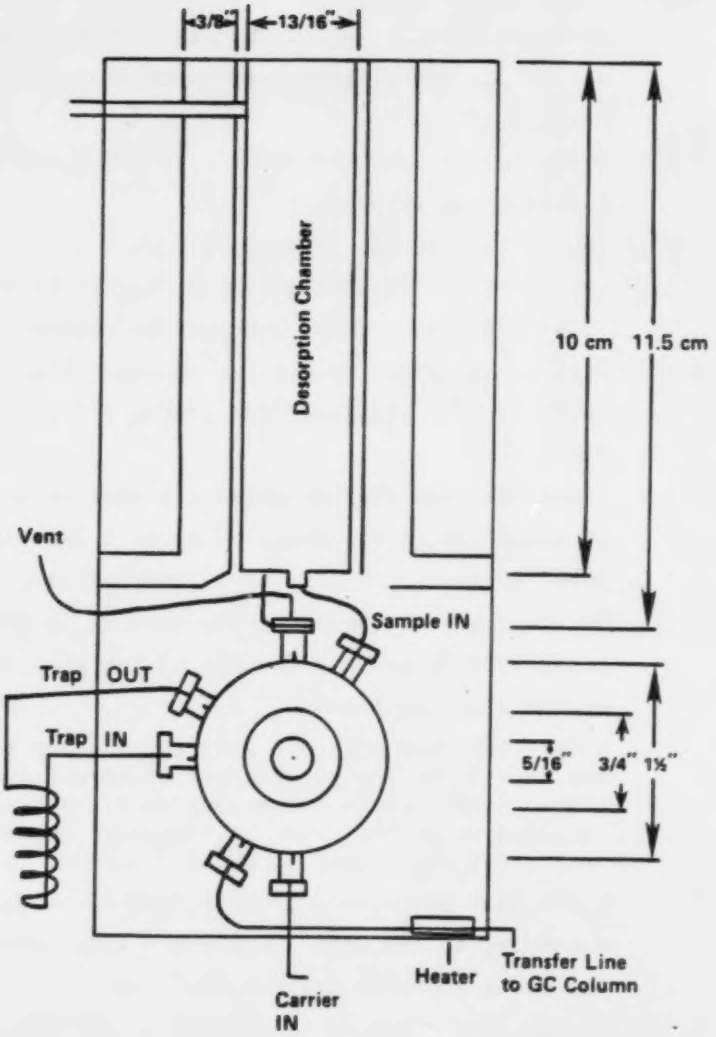

3/8"
13/16"
Desorption Chamber
10 cm
11.5 cm
Vent
Sample IN
Trap
OUT
Trap
IN
5/16"
3/4"
1½"
Heater
Transfer Line to GC Column
Carrier IN

Figure 2. 6-Port Valco high temperature valve and connections.

4.3.8 After inspection of all the areas indicated above, reinstall the desorption unit above the gas chromatograph oven with the transfer line going through a hole in the gas chromatograph injector port block closest to the ion source.

4.3.9 At this point do not reassemble the desorption unit.

4.4 Installation of Gas Flows to Allow Leak-Testing of the Thermal Desorption Unit

4.4.1 To inspect the desorption unit further, connect the helium purge flow.

4.4.2 Attach a 1/4"-1/8" female Swagelok pipe fitting to the regulator of the helium tank.

4.4.3 Attach a length of 1/8" copper tubing sufficient to reach from the regulator to the mass flow controller.

4.4.4 Insert a fine metering valve in this copper line near the regulator and insert an on/off toggle valve in the copper line near the desorption unit.

4.4.5 Connect the end of the line subsequent to the toggle valve to a mass flow controller.

4.4.6 Using 1/8" copper tubing, connect the flow controller to the desorption unit.

4.4.7 Set the pressure of the regulator at approximately 40 psi.

4.4.8 Open the tank valve and the regulator valve to allow helium to fill the line.

4.4.9 Regulate the flow of gas to 15-20 mL/min using the fine metering valve.

4.4.10 Verify the flow rate by attaching a bubble flow meter to the stainless steel "helium out" line of the desorption unit.

4.4.11 Label this gas line with a metal tag reading "Helium purge line."

NOTE: In order to check that the desorption unit is leak-tight in both "Desorb" and "Inject" positions, it is necessary also to connect the carrier gas.

4.4.12 Connect a 1/4"-1/8" female Swagelok pipe fitting to a helium tank and set the regulator to a pressure reading of 40 psi.

4.4.13 Connect a length of 1/8" copper tubing to this fitting and label "Carrier Gas" with a metal tag.

4.4.14 On the Finnigan 3300, connect the carrier gas line to the low flow head of the Tylan Helium Mass Flow Controller.

4.4.15 Attach the copper tubing from the flow controller to the "Helium in" line of the six-port valve.

4.4.16 Open the valve of the helium tank regulator.

4.4.17 Turn on the flow controller.

4.4.18 Set the black knob to the number 1 position.

NOTE: Once the POWER of the flow controller is ON, it is essential to keep a flow of helium through the controller. Leaving the flow controller ON without a flow of helium will result in burning out the regulator heads of the flow controller.

4.4.19 Adjust the black grooved knob centered on the front panel of the flow controller until the digital readout is 1.0 mL/min.

4.4.20 Verify this reading with a bubble flow meter to ensure that the flow controller is working properly.

4.5 Leak Testing of the Thermal Desorption Unit

4.5.1 Check the six-port valve for leaks in the connections after the gas flows have been connected.

4.5.2 Check the valve in both the DESORB and INJECT positions.

4.5.3 Cap off the transfer line and vent line so that no gas flow can exit from the valve.

4.5.4 Turn the handle of the valve to the DESORB mode (up).

4.5.5 Turn up the flow controller to approximately 5.0 mL/min.

NOTE: The reading on the flow controller will remain at 5.0 mL/min for approximately 1 to 2 minutes and should then drop quickly. If the digital readout of the flow controller does not drop to zero or a negative number, either the body of the valve, the connections to the valve, or the Teflon cap of the desorption unit (or some combination of the three) is leaking.

4.5.6 Examine the final reading of the flow controller to determine the severity of the leak.

4.5.7 Inspect all connections going into the desorption unit and six-port valve for leaks by putting Snoop on the connections.

NOTE: Bubbles will appear immediately at a leaking connection.

4.5.8 Allow the Snoop to remain on the fittings under observation for several minutes because a slow leak may be present.

4.5.9 Tighten the appropriate fitting(s) if a leak is found.

4.5.10 Recheck the unit by repressurizing and following the above leak-checking sequence.

NOTE: If tightening a fitting does not produce a leak-tight unit, the nut and ferrule may not be seated properly. Loosen the nut and remove the line from the six-port valve. Cut the line behind the nut so that a new ferrule can be installed and seated. If this cutting will result in a line which will be too short to use after cutting, it will be necessary to use a new length of nickel tubing.

NOTE: Do not use a tubing cutter on nickel tubing. The tubing cutter will deform or even partially seal the severed line in the process of cutting, leaving an opening with a greatly reduced diameter which will act as a restrictor to gas flow. Cut the tubing by scoring on one side with a file and snapping off the desired length of tubing.

NOTE: Inspect the nut cut from the line carefully in order to be certain that the threads are in sufficiently good condition to allow reuse. If this nut cannot be reused, obtain a new nut in order to obtain an effective seal.

4.6 Seating Ferrules

4.6.1 Since the use of a new valve to seat a ferrule could result in a nut breaking off inside the valve and rendering it unfit for use, seat a ferrule in an old valve or other appropriate fitting.

4.6.2 Place a 1/16" Valco nut and ferrule on the metal line.

4.6.3 Insert the line into a threaded opening in the old valve or fitting.

4.6.4 Tighten the nut with a 1/4" wrench.

4.6.5 Verify that the ferrule is seated by checking whether the ferrule moves on the line: a properly seated ferrule will not move.

4.6.6 When the ferrule has been seated correctly, reinstall the line on the six-port valve and repeat the leak-checking procedure.

4.7 Leak-Testing the Teflon Cap

4.7.1 Inspect the Teflon cap of the thermal desorption unit for leaks with the valve set in the DESORB mode.

4.7.2 Apply Snoop on top of the cap as well as around it.

4.7.3 If a leak is not observed instantaneously, leave the Snoop on for several minutes to check for a slow leak.

4.7.4 If a leak is found, replace the Teflon cap with a new one and repeat the leak-checking procedure.

4.8 Replacing the Teflon Cap of the Thermal Desorption Unit

4.8.1 Remove the two screws attached to the thermal desorption chamber which hold the cap assembly on the desorption unit with a flat-bladed screwdriver.

NOTE: The entire cap handle can now be removed.

4.8.2 Inspect underneath the Teflon cap to verify the presence of a brass disk with a spring soldered to a screw.

4.8.3 Loosen the screw to allow the cap and washer to be removed.

4.8.4 Center the washer on the new Teflon cap (25 mm o.d. x 5 mm thick).

4.8.5 Screw the spring soldered to a screw back onto the unit.

4.8.6 Put the handle assembly back on top of the thermal desorption unit.

4.8.7 Tighten the two side screws with a flat-bladed screwdriver.

4.8.8 Center the washer and cap on the top of the thermal desorption unit.

4.8.9 Push the handle down to allow the cap and desorption chamber to be flush with each other.

4.8.10 Repeat the leak-checking procedure.

4.8.11 If the cap does not now seal tightly, install another small washer above the washer already on top of the cap.

4.8.12 Repeat the leak-checking procedure.

4.8.13 If the cap still does not seal tightly, repeat the procedure to install a new cap.

4.8.14 Repeat the leak-testing procedure.

4.9 Reassembly of the Desorption Unit

4.9.1 When all connections to the valve and the Teflon cap of the thermal desorption unit have been inspected and no leaks have been found to be present, reassemble the thermal desorption unit.

4.9.2 Screw the back half of the aluminum block into place using a 5/32" Allen wrench on the four bolts.

4.9.3 Recheck the thermocouples for the block to verify that they are securely in place.

4.9.4 Check again to be sure that the thermocouples are insulated by glass capillary tubes on their metal ends.

4.9.5 Insert the insulation around the aluminum block (with gloves on) being careful not to dislodge any thermocouple or heater wires.

4.9.6 Connect the back panel to the thermal desorption unit.

5.0 ADJUSTMENT OF GAS FLOWS

The flows for the helium purge and helium carrier gas have already been adjusted to 15.0 mL/min and 1.0 mL/min, respectively, in order to test the thermal desorption unit for leaks. The other gas flow needed to operate the thermal desorption unit is a nitrogen flow into the nickel trap.

5.1 Materials and Equipment

The materials and equipment needed to set up and adjust the gas flows for operation of the thermal desorption unit are listed below.

5.1.1 Nitrogen (1 tank) (National Welders)
5.1.2 Nitrogen regulator (Matheson)
5.1.3 1/4"-1/8" female pipe fitting (Swagelok)
5.1.4 1/8" copper tubing (several feet) (Dillon Supply)
5.1.5 1/8" nuts and ferrules (4) (Swagelok)
5.1.6 1/4" copper tubing (approximately 1 foot) (Dillon Supply)
5.1.7 1/4" nuts and ferrules (2) (Swagelok)
5.1.8 1/4" to 1/8" reducing unions (2) (Swagelok)
5.1.9 ON/OFF toggle valve (Raleigh Valve and Fitting)
5.1.10 Dewar flask (Fisher)

5.2 Assembling Gas Lines and Setting Flows

5.2.1 Attach a 1/4" to 1/8" female pipe fitting to the regulator of the nitrogen tank.

5.2.2 Set the regulator of the nitrogen tank to read a pressure of approximately 60-70 psi.

5.2.3 Attach an 1/8" copper line to the pipe fitting with 1/8" nuts and ferrules.

5.2.4 Put a toggle valve into the copper line within easy reach of the thermal desorption unit.

5.2.5 Attach the 1/8" copper line to the 1/4" copper line.

5.2.6 Allow two or three coils of the 1/4" copper line to rest at the bottom of the Dewar flask.

5.2.7 Attach the 1/4" copper line to the 1/8" copper line.

5.2.8 Terminate the copper tubing at the nickel trap (see Figure 3).

6.0 ADJUSTMENT OF DESORPTION UNIT TEMPERATURES

All temperatures within the thermal desorption unit must be monitored very closely during their initial heat-up period to verify that the potentiometers on the control unit are adjusted correctly and that the thermocouples within the desorption unit are functioning properly.

6.1 Temperature of the Desorption Unit Aluminum Block

6.1.1 After the flow of helium to the six-port valve is established, heat the aluminum block to 270°C.

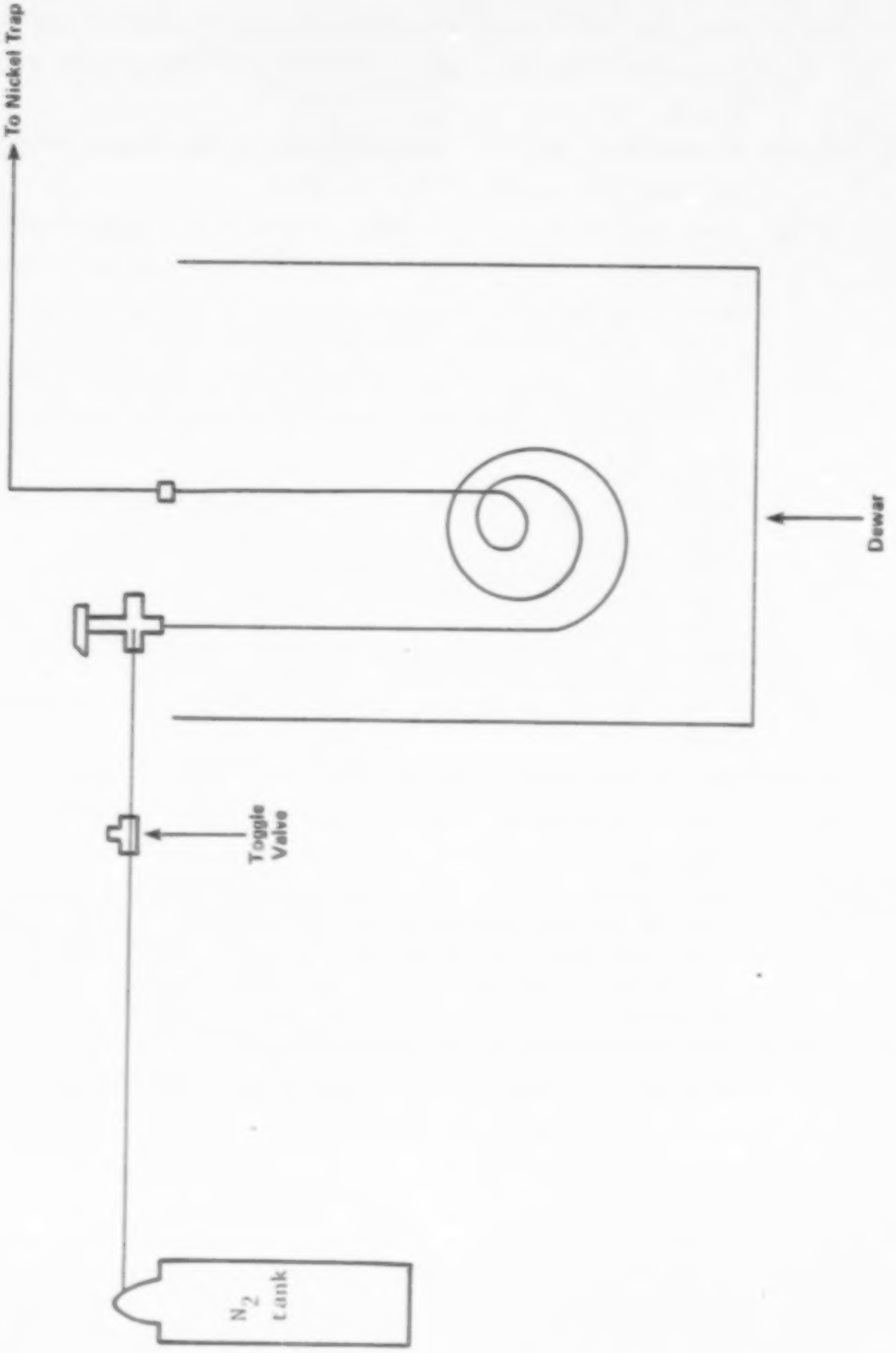

To Nickel Trap
Dewar
Toggle Valve
N_2 tank

Figure 3. Nitrogen gas flow.

6.1.2 To heat the aluminum block, turn on the toggle switch labeled "Desorption Unit Block" located on a metal panel beneath the flow controller, above the gas chromatograph.

6.1.3 Turn the metal knob beside the digital readout panel to the first position in order to monitor the temperature of the block.

6.1.4 Verify that the red light adjacent to the toggle switch is lit when the toggle switch is on.

6.1.5 Turn the black potentiometer clockwise to increase the temperature and counterclockwise to decrease the temperature.

NOTE: An orange light above the black potentiometer will be lit when the aluminum block is heating.

6.1.6 While the block is heating, rotate the six-port valve frequently from the DESORB mode to the INJECT mode and back again.

NOTE: The valve should rotate smoothly at 270°C.

NOTE: If the valve is not rotated during heating, it will most probably freeze and will not be able to be rotated at all. If the valve freezes, cool the aluminum block to room temperature and try gently to rotate the valve. If the valve remains frozen, disassemble the thermal desorption unit completely, remove the frozen valve, install a new valve, and repeat the leak-checking and reassembly procedures.

6.2 Adjustment of the Temperature of the Gold-Plated Transfer Line

6.2.1 While the aluminum block is heating, heat the gold-plated transfer line from the six-port valve to 240°C.

NOTE: The controls for this transfer line are located and labeled on the same panel as the controls for the aluminum block and the temperature of the gold-plated transfer line can be monitored by turning the metal knob to the third position.

6.3 Temperature Regulation of the Nickel Trap

6.3.1 Monitor the temperature of the nickel trap in the second position of the metal knob.

6.3.2 Set the potentiometer so that the heating is halted at 240°C.

6.3.3 Allow the trap to go through at least one heating and cooling cycle to verify that it is functioning properly.

NOTE: Often, even when the heating is manually turned off at 240°C, the nickel trap will overshoot to approximately 255°C.

7.0 INSTALLATION OF FUSED SILICA CAPILLARY COLUMNS

Capillary gas chromatography is used in the analysis of organic compounds desorbed from Tenax cartridges in order to attain the maximum sensitivity and resolution.

7.1 Materials and Equipment

7.1.1 50 or 60 m wide-bore fused silica capillary column, either thick film coating of phase or chemically bonded (American Scientific Products)

7.1.2 1/4" open-end wrenches (2) (Sears)

7.1.3 0.4 mm i.d. graphite ferrule (2) (Supelco)

7.1.4 0.4 mm i.d. graphite/Vespel ferrule (1) (Supelco)

7.1.5 1/16" stainless steel back ferrule (2) (Raleigh Valve and Fitting)

7.1.6 1/16" stainless steel front ferrule (1) (Raleigh Valve and Fitting)

7.1.7 Zero dead volume fitting (1) (Swagelok)

7.1.8 1/16" threaded male nut (2) (Swagelok)

7.1.9 Old 10 microliter syringe (Supelco)

7.1.10 3 cm o.d., 1.5 cm thick high temperature septum (Supelco)

7.1.11 1/16" stainless steel female nut (Swagelok)

7.1.12 5/16" open end wrench (Sears)

7.2 Procedure

7.2.1 Verify that a 1/16" Swagelok male nut with 1/16" back and front ferrule can be slipped over the gold-plated nickel transfer line from the six-port valve.

7.2.2 If the fittings cannot be slipped over the transfer line, carefully file the end of the line so the fittings can be slipped over it.

7.2.3 Attach the zero dead volume fitting, being certain to seat the ferrule properly.

7.2.4 Remove one loop of the fused silica capillary column from the closed side of the column cage.

7.2.5 Remove three loops from the open side.

7.2.6 Remove the cover from the transfer line oven.

7.2.7 Feed a length of column equivalent to three loops through a small opening at the rear of the oven of the gas chromatograph.

7.2.8 Pull the column end up into the transfer line oven.

7.2.9 Put the column cage on the metal column holding bar in the oven of the gas chromatograph.

7.2.10 In this order, carefully put on the front end of the column: 1/16" male threaded nut, 1/16" stainless steel back ferrule, 3 cm o.d. high temperature septum, and a 0.4 mm i.d. graphite ferrule.

NOTE: The order is critical to seal the column properly in the zero dead volume fitting (see Figure 4).

7.2.11 If the column cannot be pushed through the opening in the ferrule, bore out the graphite ferrule with an old 10 microliter syringe needle.

7.2.12 Once the graphite ferrule is on the end of the column, break off about 2-3 inches of column to ensure that no graphite ferrule particles have blocked the opening of the column.

7.2.13 Be sure that the break is clean with no rough edges.

7.2.14 Position and hold the column with one hand while sliding the nut up to the zero dead volume fitting.

7.2.15 Thread and tighten the nut while holding the column in position with the other hand, holding the column until the nut is finger-tight, at which time the column will stay in position without being held.

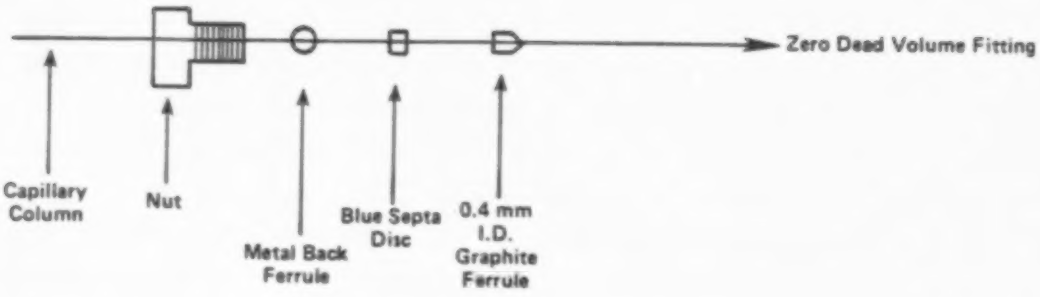

Zero Dead Volume Fitting
Capillary Column
Nut
Metal Back Ferrule
Blue Septa Disc
0.4 mm I.D. Graphite Ferrule

Figure 4. Front end of GC column.

7.2.16 Continue tightening the nut with two 1/4" open-end wrenches, one wrench holding the zero dead volume fitting and the other turning the nut.

7.2.17 Tighten the nut until it feels snug.

7.2.18 Before connecting the back end of the column, check the helium carrier gas flow to verify that helium is flowing through the column.

NOTE: If the column has not been conditioned, do not connect the back end of the column. Follow the column manufacturer's directions for conditioning the column, and connect the back end only after the column has been conditioned.

7.2.19 Check for gas flow through the capillary column by filling a small vial with water and putting the back end of the column into the water.

NOTE: If flow is present, bubbles will be observed.

7.2.20 If no bubbles are observed, verify that the helium tank valve is open, the flow controller is turned on and the flow controller is set at 1.0 mL/min.

7.2.21 If there is still no flow, check whether the column has broken off inside the zero dead volume fitting.

7.2.22 Once flow through the column has been established, put a 1/16" stainless steel female nut with 0.4 mm i.d. graphite/Vespel ferrule on the back end of the column.

7.2.23 Bore out the graphite/Vespel ferrule with the old syringe needle to allow the ferrule to fit over the end of the column.

7.2.24 Push the ferrule onto the end of the column.

7.2.25 Break off approximately 2 inches of the column to ensure that no particles of the ferrule have entered the column to block the flow of helium.

7.2.26 Measure 8.3 cm from the end of the column including the 1/16" stainless steel female nut (see Figure 5).

7.2.27 Holding the column with one hand, thread and finger-tighten the female nut to the threaded male fitting located in the transfer line oven on the side nearest the ion source.

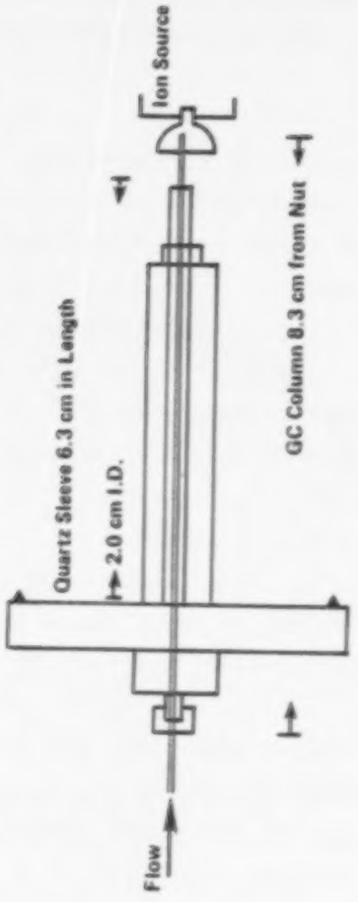

Ion Source
Quartz Sleeve 6.3 cm in Length
2.0 cm I.D.
GC Column 8.3 cm from Nut
Flow

Figure 5. Back end of GC column.

7.2.28 Tighten the nut snugly with 5/16" open-end wrench.

8.0 STARTING THE VACUUM SYSTEM OF THE MASS SPECTROMETER

When all fittings are air-tight, start the vacuum system of the mass spectrometer to produce the high vacuum essential for operation. Since it is not possible to leave the mechanical and diffusion pumps in operation during the changing/insertion of a capillary gas chromatographic column, the mass spectrometer system must be put under vacuum.

8.1 Procedure

8.1.1 To initiate operation of the vacuum system after the capillary column has been positioned in the gas chromatograph and both ends sealed, turn on the mechanical pumps by flipping up the switch on the vacuum controller labeled "FOREPUMP." (Figure 6 presents an illustration of the vacuum controller panel of the mass spectrometer.)

8.1.2 As the mechanical pumps evacuate the ion source and analyzer regions of the mass spectrometer, monitor the forepressure in the ionizer by observing the appropriate gauge below the vacuum controller panel.

8.1.3 When the ionizer forepressure reads 50 millitorr or lower, turn on the diffusion pumps to the override position making sure the cooling water to the diffusion pump is turned on.

8.1.4 When the diffusion pumps have heated for approximately 45 minutes, depress the Ionization Gauge START pushbutton in order to light the ionization gauge and enable the Vacuum Protect circuitry.

8.1.5 Calibrate the Ionization Gauge by holding the START button in and turning the Zero knob until the needle points to CAL.

8.1.6 If the ionization gauge does not remain at this point, wait 5 minutes and try again.

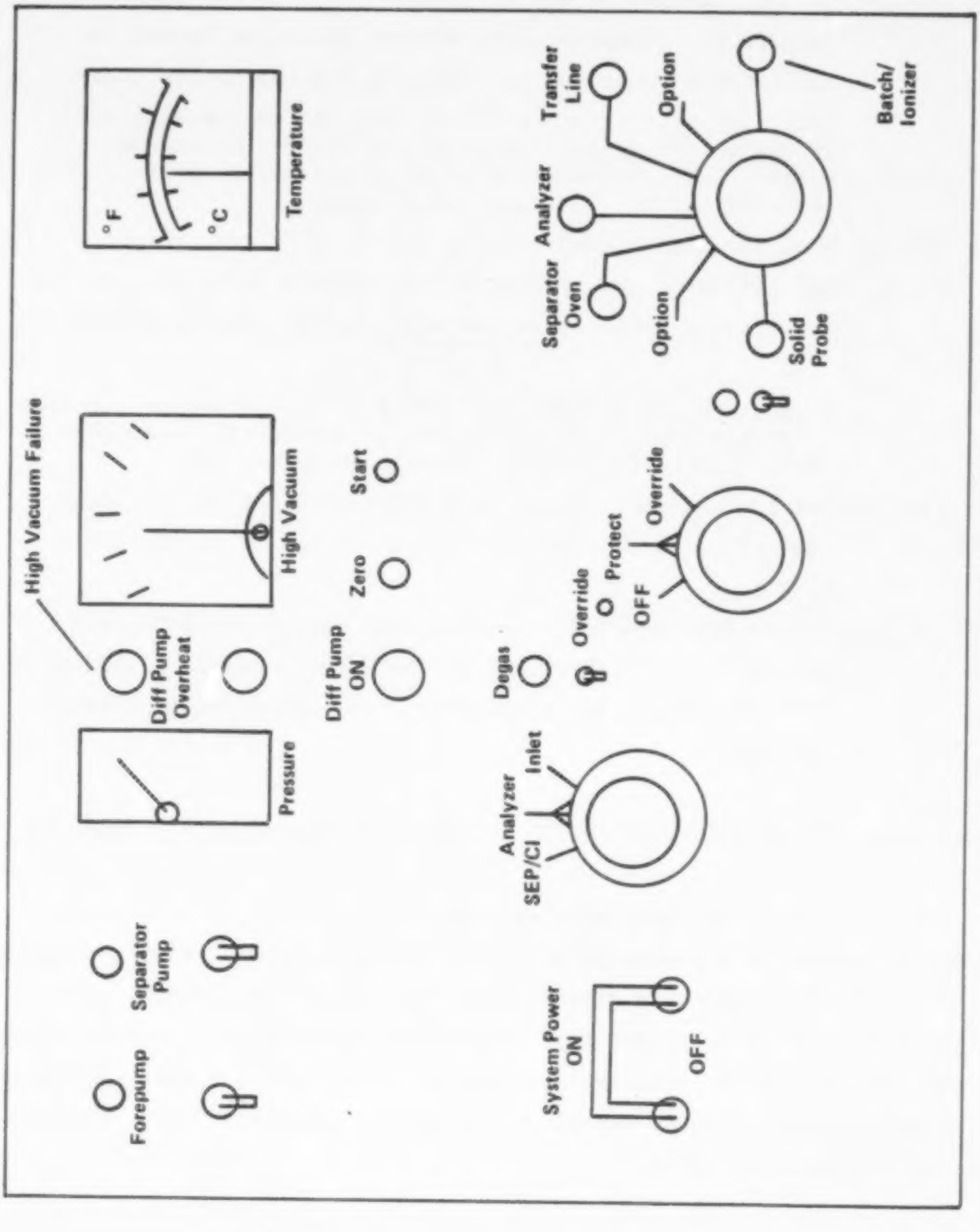

High Vacuum Failure
Diff Pump Overheat
Pressure
High Vacuum
°F
°C
Temperature
Forepump
Separator Pump
Diff Pump ON
Zero
Start
System Power ON
OFF
SEP/CI
Analyzer
Inlet
Degas
Override
OFF
Protect
Override
Separator Oven
Analyzer
Transfer Line
Option
Option
Solid Probe
Batch/Ionizer

Figure 6. Vacuum controller module front

8.1.7 If the gauge does not light after repeated efforts, use a meter to verify that the filament of the ionization gauge is continuous.

8.1.8 If the filament of the ionization gauge is not continuous, consult the Finnigan GC/MS Systems Operation Manual for the correct procedure for changing the ionization gauge.

NOTE: It will be necessary to vent the source/analyzer to change the ionization gauge, so follow the vacuum system start-up procedure as described above after installation of a new ionization gauge.

8.1.9 When the high vacuum reading is 3×10^{-5} torr, rotate the diffusion pump switch to the PROTECT position to allow power to be supplied to the spectrometer control module.

CAUTION

During operation permanent damage to the secondary electron multiplier can occur if the manifold temperature exceeds 100°C with voltage going through the multiplier.

8.1.10 Set the injector block temperature to 250°C by adjusting the appropriately labelled switch on the GC controller module.

8.1.11 Adjust the transfer line oven temperature to 250°C with the knob labeled ANALYZER on the Vacuum Controller Module, but read the actual temperature from the position labeled SEPARATOR.

9.0 STARTING THE DUAL DISK DRIVES OF THE DATA GENERAL NOVA 3 COMPUTER

The Finnigan 4021 mass spectrometer and the Finnigan 3300 mass spectrometer share a common data system, the Data General Nova 3 computer with two Perkin-Elmer disk drives, each containing a removable and a fixed disk. It is therefore imperative that the operators on both systems establish clearly who will acquire data on which disk and what procedure will be followed if it is necessary for one of the operators to change one of the removable disks.

9.1 Procedure

9.1.1 Before starting the disk drives, check the thumbwheel dial on both the top and bottom disk drive.

9.1.2 Set the top drive at ZERO and the bottom drive at TWO in order to designate the removable disks as Units 0 and 2 while the two fixed disks are Units 1 and 3.

9.1.3 With disks present in both drives, depress the power switch on both drives.

9.1.4 Wait for the LOAD light to come on.

9.1.5 Be sure that the PROT CART and PROT FIXED switches on each drive are depressed in order to protect the disks against damage.

9.1.6 Flip the RUN/LOAD switch to RUN.

9.1.7 Wait for a READY light to appear on each drive.

9.1.8 When the READY light is observed, turn off the PROT CART and PROT FIXED switches; the disks are now ready for operation.

10.0 STARTING COMPUTER TERMINALS ON THE DATA GENERAL NOVA 3

The Finnigan 4021 and the Finnigan 3300 each have a Tektronix 4010 computer terminal which is routinely turned OFF at the end of the operating day.

10.1 Procedure

10.1.1 To prepare the terminal for operation, depress the MAIN POWER switch located beneath the terminal keyboard on a gray panel.

10.1.2 Verify that a red light labeled POWER on the keyboard panel lights when power is supplied to the terminal.

10.1.3 Check to be sure that the LOCAL/LINE switch on the terminal keyboard is in the LINE position.

11.0 STARTING THE FINNIGAN 3300 MASS SPECTROMETER

In routine operation, the mass spectrometer filament and electron multiplier are turned OFF at the end of the operating day; all other temperatures and systems controls are left in operation.

11.1 Operation of the Spectrometer Control Module

11.1.1 Turn on the FILAMENT and MULTIPLIER on the spectrometer control module (see Figure 7).

11.1.2 Set the emission current of the filament at 0.5 mA; adjust to the correct value with the knob labeled EMISSION CURRENT.

11.1.3 Turn the multiplier knob to the 2 kV position.

11.1.4 Adjust for the desired multiplier setting with the knob labeled FINE.

NOTE: The present setting for the multiplier is approximately 1800 volts, but the setting at any given time is a function of the age of the multiplier and other mass spectrometer system parameters.

11.1.5 Read the values for the emission current and the multiplier voltage by using the METER FUNCTION switch.

11.2 Tuning of the Radio Frequency (RF)

11.2.1 Turn the Scan Range knob on the spectrometer control module fully counterclockwise.

11.2.2 Adjust the first mass control knob to mass 100.

11.2.3 Obtain a voltmeter.

11.2.4 Place the black test lead in the black hole on the back of the spectrometer control module chassis.

11.2.5 Insert the red test lead in the orange hole.

11.2.6 Verify that the internal/external mode switch on the front of the chassis is in the internal mode.

11.2.7 Set the voltmeter on three volts.

NOTE: There should be a voltage reading.

11.2.8 Using a flat-bladed screwdriver, turn the metal knob labeled TUNING until the lowest possible voltage reading occurs.

11.2.9 Repeat these steps at mass 500 and 1000.

11.2.10 Repeat this procedure any time RF tubes are replaced, the ion source is cleaned, or the electron multiplier is replaced.

11.3 Setting the Zero of the Mass Spectrometer

11.3.1 To zero the mass spectrometer, turn on the multiplier.

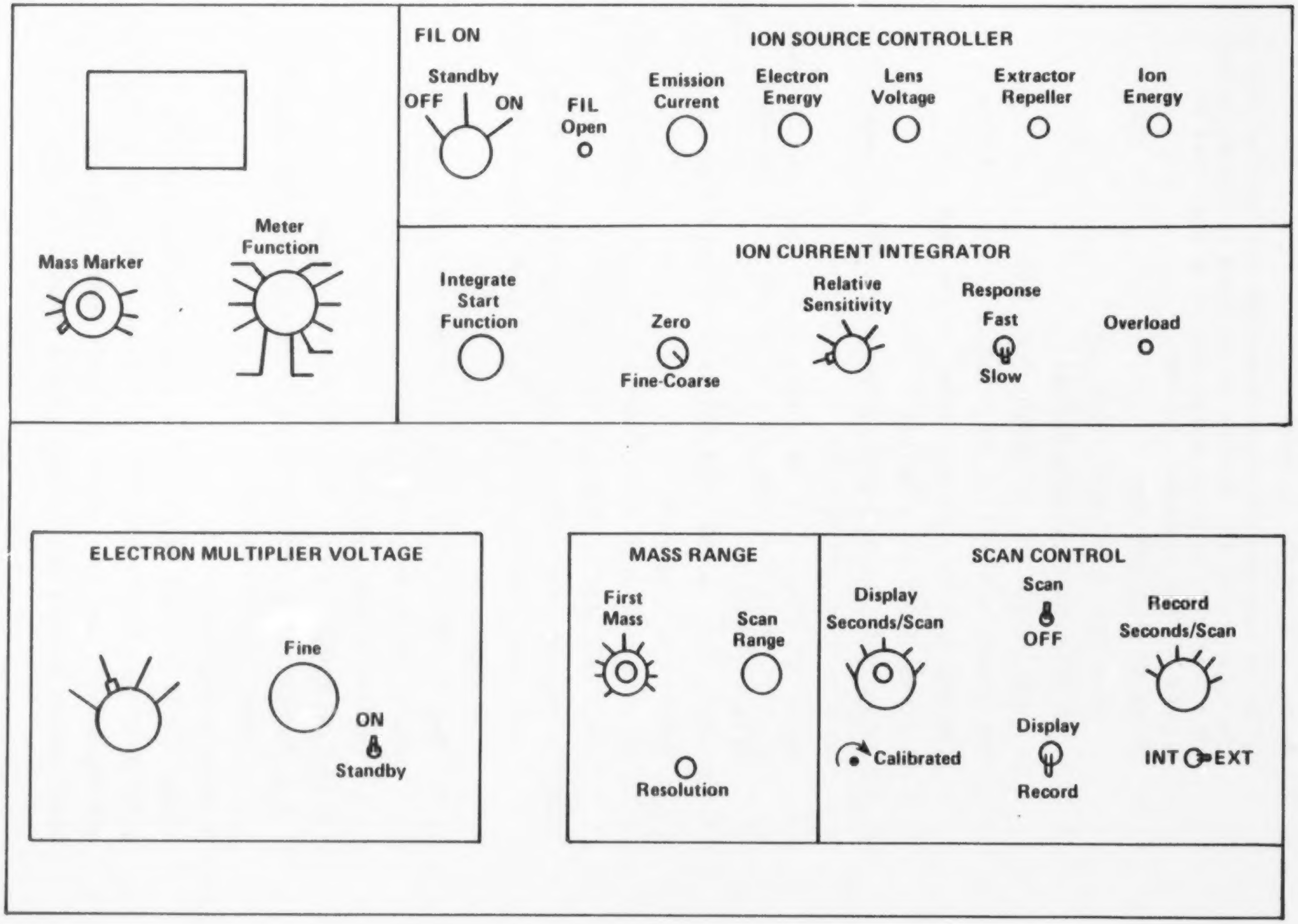

FIL ON
ION SOURCE CONTROLLER
Standby
OFF
ON
FIL Open
Emission Current
Electron Energy
Lens Voltage
Extractor Repeller
Ion Energy
Meter Function
Mass Marker
ION CURRENT INTEGRATOR
Integrate Start Function
Zero
Fine-Coarse
Relative Sensitivity
Response
Fast
Slow
Overload
ELECTRON MULTIPLIER VOLTAGE
Fine
ON
Standby
MASS RANGE
First Mass
Scan Range
Resolution
SCAN CONTROL
Display Seconds/Scan
Calibrated
Scan
OFF
Display
Record
Record Seconds/Scan
INT
EXT

Figure 7. Spectrometer control module front panel.

11.3.2 Set the SCCPE MODE switch in the External position.

11.3.3 Do not turn on the filament.

NOTE: A computer procedure named ZMS has been written to aid the operator in the zeroing of the mass spectrometer and this procedure is permanently stored on disk Unit #1. A copy of this procedure follows:

```
TRACE OF PROCEDURE ZMS
*  SCAN 3300 (300;400;1.9;0;0;.1)
*  ACQU/O ZMS(K1;T1;M0;G1000;E)
*  MAP/C ZMS(G;360,370;V10;D1,1000,200)
```

11.3.4 With power supplied to the computer terminal, type a CONTROL D and a > should appear on the screen of the visual display terminal.

11.3.5 To activate the zeroing procedure type ZMS followed by a carriage return <CR>.

NOTE: At this point the computer will show ions 360,362, ... 370 under the MAP/C program of the Finnigan Incos Data System.

11.3.6 Verify that random noise spikes are observed at these masses (see Figure 8).

11.3.7 If no noise spikes are observed or a great number appear, reset the zero of the mass spectrometer.

11.3.8 To regulate the number of the noise spikes, adjust the Zero Control knob located on the Output Module Front Panel (see Figure 9).

11.3.9 When the MAP/C display produces random spikes at the masses displayed, type a CONTROL D to regain operating control of the computer.

11.3.10 Type E <CR> to exit from the MAP/C program.

11.3.11 Terminate the ZMS procedure by typing STOP <CR> when a caret (>) is observed.

11.4 DC Balance Adjustment

Adjust the DC Balance of the rf/dc generator each time the quadrupole mass filter assembly is cleaned. The test points and adjustments are located on the RF/DC board inside the Spectrometer Control Module electronics chassis.

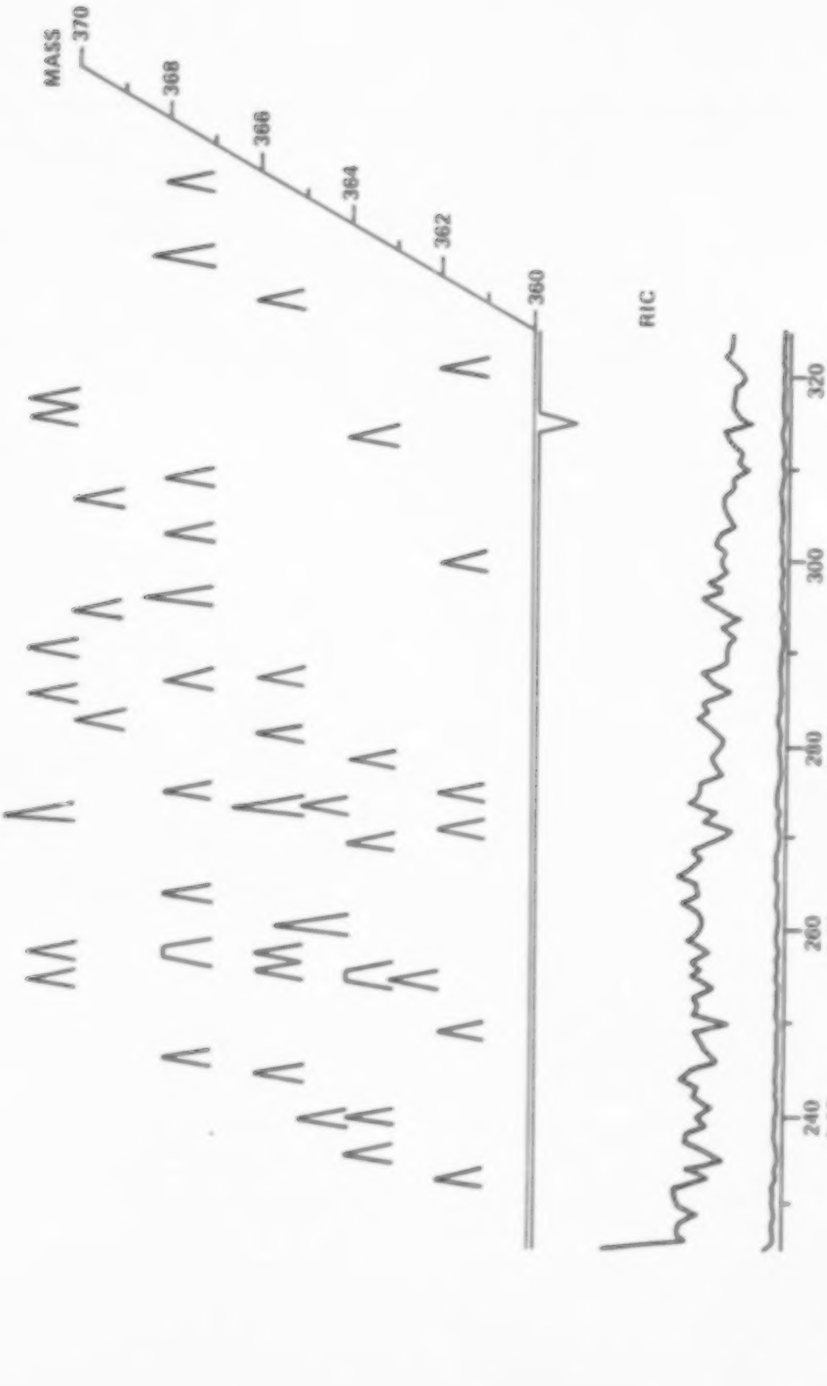


Figure 8. MAP/C with random spikes in ZMS.

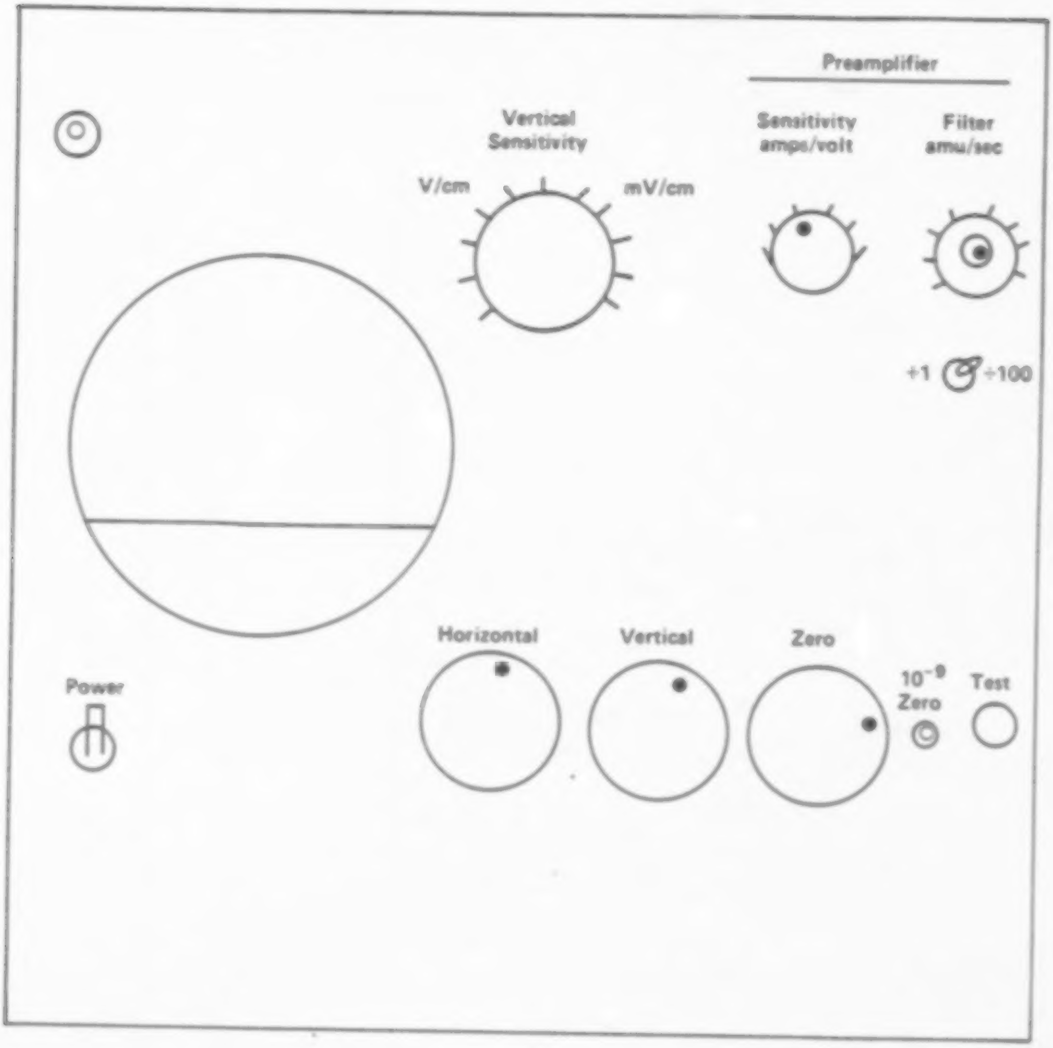

Preamplifier
Vertical Sensitivity
V/cm
mV/cm
Sensitivity amps/volt
Filter amu/sec
+1
÷100
Horizontal
Vertical
Zero
10^{-9} Zero
Test
Power

Figure 9. Output module front panel.

11.4.1 Set the following controls on the Spectrometer Control Module to the following positions:
SCAN/OFF switch - SCAN
SCAN RANGE CONTROL - fully clockwise
FIRST MASS CONTROL - fully counterclockwise
MODE switch - internal

11.4.2 Set the NORM/TEST switch on the back of the Output Module to the TEST position.

11.4.3 Connect the test probe to the TEST connector located on the front panel.

11.4.4 Center the oscilloscope trace in the middle of the oscilloscope using the VERTICAL and HORIZONTAL adjustment knobs.

11.4.5 Connect one lead of the test probe to the TP4 connector and the other test probe lead to the TP5 connector located on the RF/DC control board.

11.4.6 Set the scope sensitivity control knob to a setting of 20 mV/cm.

11.4.7 Adjust the DC balance potentiometer on the RF/DC Control board so the trace on the oscilloscope is horizontal.

11.4.8 Reset the NORM/TEST switch back to the NORM position after this procedure is finished.

12.0 INTRODUCTION OF PERFLUOROTRIBUTYLAMINE (FC-43) CALIBRATION GAS

In order to calibrate the mass spectrometer/computer system so that the correct masses will be assigned, it is necessary to introduce perfluorotributylamine (FC-43).

12.1 Filling the Molecular Leak System

12.1.1 Insert the molecular leak inlet system through the hexagonal black knob until it touches the closed analyzer valve.

12.1.2 Tighten the knob, open the analyzer valve and inlet system valve.

12.1.3 Allow the inlet to pump out.

12.1.4 Verify that the filament and electron multiplier are turned off.

12.1.5 Inject approximately 0.2 µL of FC-43 into the inlet system with the inlet system valve closed.

12.1.6 Push the inlet system into the analyzer and stop approximately 1/4" from the end.

12.1.7 Open the inlet system valve.

12.1.8 Turn on the filament and multiplier with the mode switch set to INTERNAL.

NOTE: FC-43 ions should appear on the scope when the mass range is between 10 and 650.

13.0 TUNING THE MASS SPECTROMETER USING FC-43

The Finnigan 3300 mass spectrometer is tuned using a group of low and high mass potentiometers and lens, extractor, and ion energy voltage adjustments. These voltages must be adjusted to produce the greatest peak amplitude consistent with good peak shape. All controls pertinent to the tuning process are located on the Ion Source Controller panel of the Spectrometer Control Module (see Figure 7). FC-43 is ideal for tuning since it has a good distribution of low and high mass ions.

13.1 Procedure

13.1.1 Set all controls on the Ion Source Controller Panel to an initial setting as listed below:

Filament Power OFF/STBY/ON - ON
Emission Current - 0.50 mA
Electron Energy - 70 eV
Lens Voltage - -100 V
Extractor Repeller - 5 to 15 V
Ion Energy - 2.2 with scan range set to zero

13.1.2 Adjust the scan range fully clockwise.

13.1.3 Turn the resolution potentiometer to its mid-position (14 turns from the end of the potentiometer).

13.1.4 Verify that FC-43 has been injected into the system and begin the initial resolution adjustments.

13.1.5 Rotate the HI MASS THRES, DET COMP, and LOW MASS COMP potentiometers located on the RF/DC control board inside the chassis fully counterclockwise.

13.1.6 Turn the LOW MASS COMP 13 turns clockwise.

13.1.7 Rotate the HI MASS RES potentiometer fully clockwise.

13.1.8 Using the FIRST MASS and Scan Range controls, find mass peaks 219 and 220 and center them on the oscilloscope.

NOTE: If no peak is found, turn up the multiplier voltage, vertical sensitivity, or both.

13.1.9 Adjust the COARSE RES potentiometer to attain the proper resolution between masses 219 and 220 (see Figure 10).

13.1.10 Find mass peaks 69 and 70 and adjust the LOW MASS COMP potentiometer for the correct resolution (see Figure 11).

NOTE: It may be necessary to adjust the lens, extractor, repeller, and ion energy voltages while adjusting the resolution in order to attain maximum signal level and acceptable peak shape. Also, the LOW MASS COMP and COARSE RES potentiometers interact with each other, thus making it imperative that the operator readjust these potentiometers until a balance occurs between the two.

13.1.11 After finding mass peaks 16, 17, 18 and 20, adjust the DET COMP potentiometer for proper resolution (see Figure 12).

13.1.12 Begin the resolution adjustment for the high mass peaks by setting the FIRST MASS control to 200 and the MASS RANGE control to 800.

13.1.13 Rotate the HI MASS THRES potentiometer clockwise.

13.1.14 Observe the trace on the oscilloscope.

13.1.15 Continue rotating the potentiometer until this trace reaches mass 264 (see Figure 13).

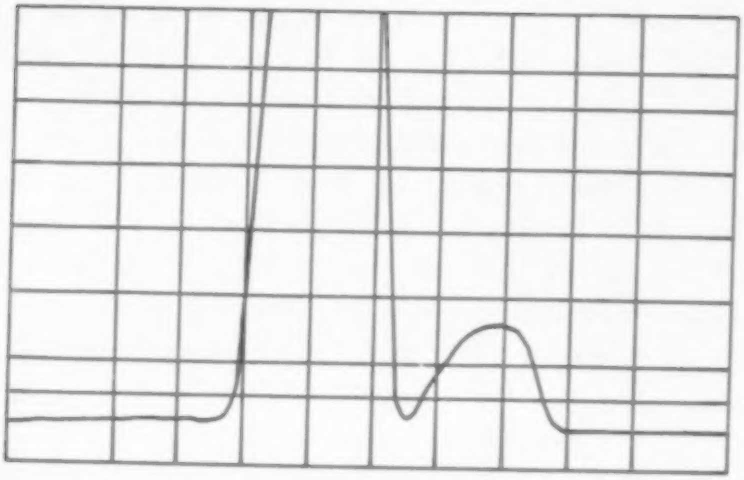


Figure 10. Mass peaks 219 and 220.

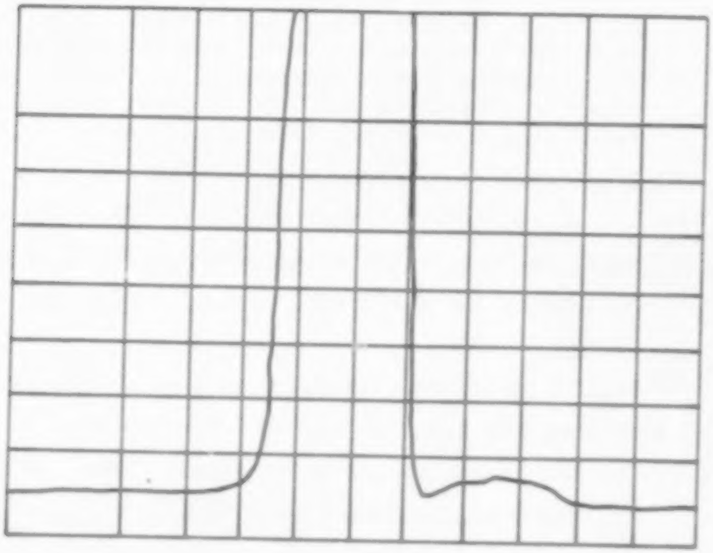


Figure 11. Mass peaks 69 and 70.

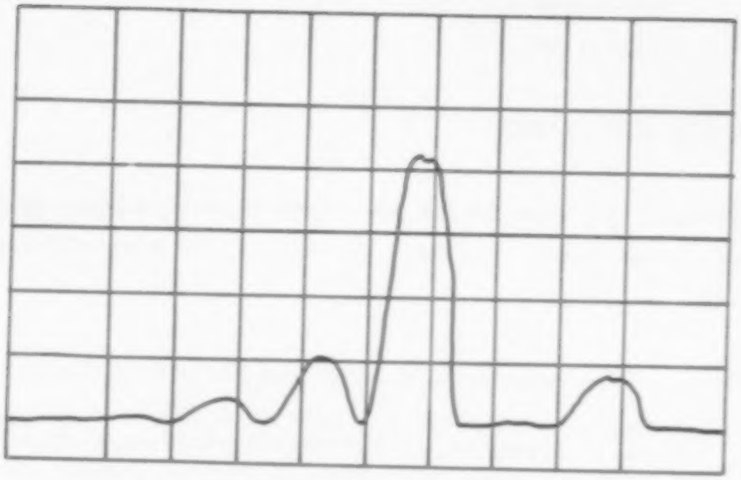


Figure 12. Mass peaks at 16, 17, 18 and 20.

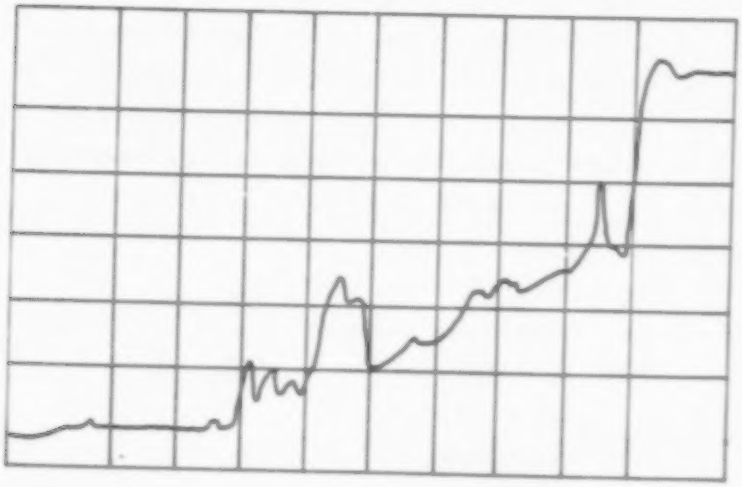


Figure 13. High mass range compensation threshold.

13.1.16 Slowly rotate HI MASS RES potentiometer until the trace on the oscilloscope diminishes to spectral lines.

13.1.17 Find peaks 502 and 503 and further adjust the HI MASS RES potentiometer until the proper resolution is observed (see Figure 14).

14.0 ACQUISITION OF DATA

The mass spectrometer is now ready to scan a mass spectrum of the calibration compound. The data system must be prepared for the acquisition of data.

14.1 Setting Scan Parameters for the Acquisition of Data

NOTE: Before acquisition of data from the mass spectrometer can occur, it is essential to set the computer and interface to scan the appropriate mass range for the correct length of time.

14.1.1 In order to initiate computer-controlled scanning of the mass spectrometer, type >SCAN 3300 <CR>.

NOTE: A series of questions will follow. The computer will have numerical values as the answers to these questions which will correspond to the last set of parameters used by the data system. These numerical values may or may not correspond to the values desired for calibration.

14.1.2 If the values presented by the computer are other than the values desired, reset the values by typing the desired value after the value presented by the computer.

```
MASS RANGE (AMU)
LOW:  20 <CR>
HIGH: 650 <CR>
SCAN TIME (seconds)
UP:  1.90 <CR>
DOWN:  0.00 <CR>
HOLD TIME (seconds)
TOP:  0.00 <CR>
BOTTOM:  0.10 <CR>
```

NOTE: These scan parameters are used in all acquisition of data for instrument calibration using FC-43.

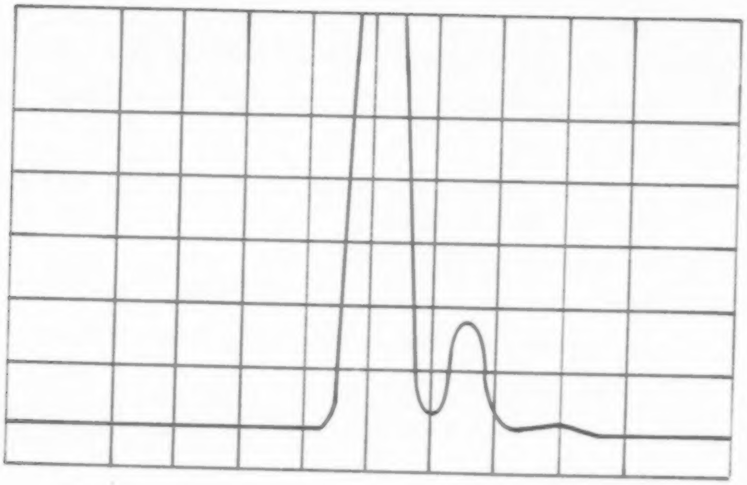


Figure 14. Mass peaks 502 and 503.

14.2 Setting Time Parameters for the Acquisition of Data

NOTE: Before acquisition of data can occur the computer clock must be set.

14.2.1 In order to set the computer clock type

>TIME <CR>.

NOTE: A series of questions will follow. The computer will have no numerical values for any of these questions (unless time has already been set). The numerical value for the hour of day must be set as "army time".

14.2.2 Insert the numerical values between the limits the system specifies for month, day, year, hour, minute, and second.

NOTE: A value must be entered for each question; a <CR> is not adequate.

14.3 Initiation of Data Acquisition

14.3.1 Before initiating acquisition of data from the mass spectrometer, set the mode switch on the spectrometer control module to EXTERNAL.

14.3.2 Start the data acquisition by typing >ACQU disk#: file name, inst <CR>.

NOTE: For example, to initiate data acquisition on disk Unit #2 for file #3233 type >ACQU 0:3233,3300 <CR>. The program ACQU will then display a summary of the state of the data acquisition and if the computer is ready to acquire data, READY TO ACQUIRE will be observed in the top right position of the terminal screen.

14.3.3 Check the display of the summary to ensure that the scanning parameters, disk unit number, file names, and all other parameters are correct.

14.3.4 Type A <CR> to obtain the set of questions which allow the entry of accounting information and operator description of the data to be acquired.

14.3.5 To respond to the computer prompts for the questions, enter Sample Name, Operating Conditions, Compound Formula (if applicable), Compound Molecular Weight, Submitter, Account Number, and Analyst.

14.3.6 If a question does not apply for a particular analysis, type <CR> and the next question will be displayed.

NOTE: After the last question has been answered, the ACQU summary table will display all the entries made for the accounting information.

14.3.7 Type K <CR> to retain the accounting information with the data file.

14.3.8 To initiate acquisition of data, type ACQU: G n <CR>, where n is the number of mass spectral scans desired.

14.4 Preparation of a Calibration Table

In order to obtain accurate mass assignments, the calibration table must be up-to-date before any analysis of samples is initiated. A calibration table is made by comparing time/intensity data acquired from scans of FC-43 with a mass/intensity reference table of FC-43 stored on disk Unit #1 on the Incos data system.

14.4.1 Acquire 15 to 20 scans of FC-43 to begin preparation of a calibration table.

14.4.2 After the acquisition of data is complete, type >CALI disk #: file name # scan number, reftable/modifiers <CR>.

NOTE: For example, >CALI 0:3233 #7, FC-43/E <CR> will calibrate the mass spectrometer/computer system using scan #7 of file 3233 compared to the FC-43 reference table and place the calibration table generated on disk unit #0.

14.4.3 Use the /E to eliminate certain background peaks which could affect the quality of the calibration.

14.4.4 Utilize the FIT program to measure the accuracy of the calibration.

14.4.5 Type >FIT <CR> to run the FIT program.

14.4.6 Examine the RMS error graph in the display generated by the FIT program to measure the accuracy of the mass assignments.

14.4.7 If the numerical value observed on the RMS error graph is greater than 50, repeat the FC-43 tuning procedure, perform another acquisition of data, and repeat the calibration process until the numerical value observed on the RMS error graph is less than 50.

14.4.8 Obtain a mass spectrum and list of the % relative abundances of the ions of FC-43 by typing >SPEC # n <CR>.

NOTE: For example: >SPEC #7 <CR> will display the seventh scan of the FC-43 data acquisition.

14.4.9 Print a hardcopy of the mass spectrum by typing SPEC: H <CR>.

14.4.10 Obtain a listing of the % relative abundances of all of the ions of the mass spectrum by typing SPEC: L <CR>.

14.4.11 Generate a hardcopy of this list by typing LIST: H <CR>.

15.0 TUNING THE MASS SPECTROMETER TO MEET PERFORMANCE SPECIFICATIONS FOR OCTAFLUOROTOLUENE (PFT)

Control of the percent relative abundances of ions of perfluorotoluene, the compound selected as the tuning standard for the analysis of volatile organic compounds from Tenax cartridges, is essential for obtaining data of the desired quality. The relative abundances of the ions of perfluorotoluene should be reproducible within a specific range established by a historical data base from day-to-day. Tuning criteria set for the major ions of the mass spectrum of PFT are as follows:

Ion	% RA of Base Peak	Range
69	28	17-39
79	8	4-12
93	16	9-23
117	46	34-58
167	16	11-19
186	66	55-77
217	100	0
236	56	46-65

Ideally the % RA should not vary by more than 10%.

15.1 Verifying the Acceptability of the Spectrum Obtained from Perfluorotoluene

NOTE: The initial tuning of the Finnigan 3300 mass spectrometer to the manufacturer's criteria for an acceptable tune for the calibration compound, FC-43, does not guarantee that an acceptable mass spectrum for PFT will be obtained.

15.1.1 Check the appearance of the mass spectrum of PFT by introducing PFT into the ion source of the mass spectrometer.

15.1.2 Pump out the heated inlet system and inject 0.2 μL PFT.

15.1.3 Introduce PFT to the ion source by the same procedure used for introduction of FC-43.

15.1.4 When PFT is present in the ion source, set the mode switch of the spectrometer control module to the "IN" position and set the First Mass Control for mass 50 and the Last Mass for mass 250.

15.1.5 Observe all the major ions of the PFT mass spectrum on the oscilloscope.

15.1.6 Set up data acquisition and acquire several scans of the PFT mass spectrum.

15.1.7 Select one scan to check the % RA.

NOTE: The spectrum of PFT obtained by introduction of PFT into the ion source using the molecular leak inlet system will differ slightly from the PFT mass spectrum obtained by thermal desorption of the PFT from a Tenax cartridge and introduction to the mass spectrometer through the GC column, but the appearance of the mass spectrum should be a very good indication of whether the tune will meet the criteria for acceptable analysis.

15.2 Adjustment of the Tune of the Mass Spectrometer to Meet PFT Tuning Criteria

15.2.1 If the mass spectrum of PFT does not meet the criteria for instrument operation, retune the instrument to meet these criteria.

15.2.2 Adjust the source voltages until the desired spectrum is obtained.

15.2.3 After any given series of adjustments, set up an acquisition and select a scan of PFT to check the tune.

15.2.4 Repeat the adjustment/acquisition process as many times as necessary in order to obtain an acceptable mass spectrum of PFT.

NOTE: Remember that in tuning for an acceptable mass spectrum of PFT, low mass ions such as 69 and 117 will be approximately 25% lower when observed from the thermal desorption process than when observed after introduction from the calibration gas inlet. For example, if a relative abundance of 40% for mass 69 is obtained when PFT is introduced through the calibration gas inlet, the relative abundance of this ion will be no greater than 30% when PFT is introduced to the mass spectrometer by thermal desorption. Ions of higher mass such as 186, 217, and 236 are not affected very much by the change in inlet systems. Therefore, in order to meet PFT tuning specifications when the compound is introduced by thermal desorption, the %RA of PFT ions obtained when the compound is introduced through the molecular leak inlet system should be approximately the following:

Ion	% RA
69	40
79	8
93	18
117	50
167	16
186	65
217	100
236	75

16.0 ESTABLISHMENT OF OPERATING CONDITIONS FOR THE GAS CHROMATOGRAPH

The Finnigan 3300 gas chromatograph/mass spectrometer/data system has a Finnigan 9600 Gas Chromatograph (GC) with a control panel for temperatures and GC programming conditions. The desired initial gas chromatograph oven temperature for thermal desorption is 30°C, which should be held for five minutes.

16.1 Procedure

16.1.1 Verify that the power switch of the GC main control panel (see Figure 15) is turned to the ON position.

16.1.2 Set the initial temperature of the GC for 30°C by opening the GC oven door and turning the INITIAL Temp°C knob to 30°.

16.1.3 Set the FINAL Temp°C knob for 240°C, the RATE °C/min knob to 2, and the INJECTOR °C knob to 25°C.

16.1.4 Turn the Temperature Control knob either to the INJECTORS or COLUMN setting to read their actual temperature.

NOTE: When reading the number of degrees/min that the GC is programming, multiply the setting of the RATE°C/min by a factor of two.

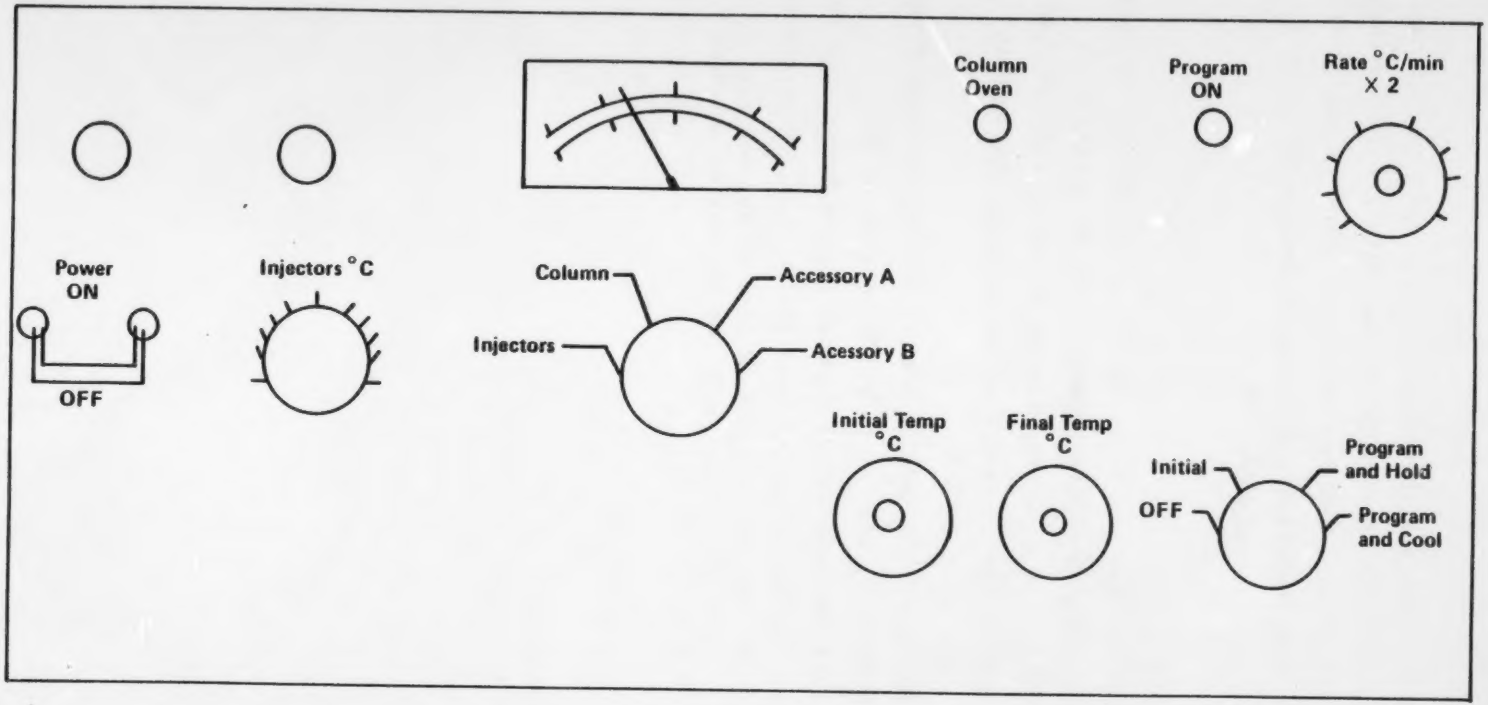

Power ON
OFF
Injectors °C
Column
Injectors
Accessory A
Acessory B
Column Oven
Program ON
Rate °C/min X 2
Initial Temp °C
Final Temp °C
Initial
OFF
Program and Hold
Program and Cool

Figure 15. Operating controls and indicators for the GC.

17.0 RECEIPT OF SAMPLES

17.1 Receive all Tenax cartridge tubes in a sealed can with the appropriate custody sheet after standards have been loaded on them.

17.2 Match the Custody Sheet with the corresponding sample to ensure no mixup has occurred.

17.3 Check each Custody Sheet carefully for the following items: 1) a signature of a person relinquishing custody; 2) the amounts of standards loaded on the cartridge; 3) the temperature and volume collected amounts; and 4) the Tenax batch number.

17.4 Do not analyze any sample that has no Custody Sheet or is missing any of the above information.

17.5 Put all cans of samples in the cartridge freezer when received.

CAUTION

Only one freezer in the Mass Spectrometry Laboratory is designated a "cartridge freezer." Failure to use this specific freezer for cartridge storage will compromise the analysis.

17.6 Log each sample in the appropriate notebook as received.

17.7 Place each Custody Sheet in the project notebook (with all other information regarding that particular sample) after signing and dating it.

17.8 Store any used cartridges in sealed cans so they can be recycled, cleaned, and used again.

18.0 DESORPTION OF A TENAX CARTRIDGE

Thermal desorption of a Tenax cartridge is accomplished by heating the Tenax cartridge in the desorption chamber while purging helium gas through it. The volatile organic compounds desorbed are then collected in the nickel capillary trap which is cooled to liquid nitrogen temperatures. This trap is heated rapidly from liquid nitrogen temperatures to 250°C and the volatilized organic compounds are injected onto the gas chromatographic column which is directly coupled to the ion source of the mass spectrometer.

18.1 Desorption Procedure

18.1.1 To initiate the process of thermal desorption of a Tenax cartridge, open the toggle valve on the nitrogen gas line and pour liquid nitrogen into the Dewar flask which contains the coiled copper tubing of the nitrogen gas line.

18.1.2 Keep the Dewar flask full or nearly full of liquid nitrogen in order to expedite the cooling process.

18.1.3 Fill the flask as often as necessary.

18.1.4 Adjust the knob of the Thermal Desorption Readout panel above the gas chromatograph to the second position in order to monitor the temperature of the trap.

18.1.5 Cool the oven of the gas chromatograph by opening the door of the gas chromatograph and setting the INITIAL TEMPERATURE to 30°C.

18.1.6 Verify that the valve of the desorption unit is in the DESORB mode (up).

18.1.7 When the temperature reading for the trap reaches -190°C and liquid nitrogen is observed to drip at a slow but steady rate from the bottom of the trap, prepare a cartridge for the desorption process.

18.1.8 Remove the sealed paint can containing the desired cartridge from the freezer.

CAUTION

Use the freezer in the laboratory designated for cartridge storage ONLY for this purpose. Inadvertent storage of containers of solvent in this freezer will result in contamination of all cartridges stored in the freezer and will compromise the analysis, since organic solvents are frequently target compounds for quantitative analysis.

CAUTION

Verify that the laboratory personnel are not involved in any process which requires the presence of open containers of organic solvents as the fumes of organic solvents will hopelessly contaminate a Tenax cartridge exposed to this atmosphere for only a few seconds, thus compromising the quantitative and/or qualitative assay.

18.1.9 Open the sealed lid of the paint can, using a flat-bladed screwdriver, beverage can opener, or other convenient tool for this purpose.

NOTE: The cartridge will be in a glass culture tube with a Teflon-lined screw cap.

18.1.10 Remove a single culture tube from the paint can and place in the wooden cartridge holder in front of the gas chromatograph.

18.1.11 Seal the paint can and replace in the freezer.

18.1.12 Release the Teflon cap of the desorption chamber.

18.1.13 Remove the cartridge from the culture tube using forceps (do not touch a cartridge with your hands).

CAUTION

DO NOT TOUCH THE CARTRIDGE WITH YOUR HANDS! The slightest trace of organic compounds present on the fingertips can be sufficient to compromise the analysis. If the cartridge is inadvertently touched, make careful note of the circumstances in both the instrument log and the project notebook.

18.1.14 Insert the cartridge immediately into the desorption chamber.

18.1.15 Close the Teflon cap of the desorption chamber.

18.1.16 Initiate the timing of the eight-minute desorption cycle.

18.1.17 Check the flow of helium through the trap frequently to verify that the helium flow rate does not slow or stop, as problems with the gas flow at this point indicate that the moisture content of the cartridge is such that the trap is freezing.

18.1.18 If the helium flow slows or stops, note this observation both in the instrument log book and in the project notebook because the results of the analysis can be severely affected as a function of the exact point in the desorption cycle at which the freezing of the trap occurred.

CAUTION

Do not continue with the desorption of a Tenax cartridge if no helium purge flow is evident after a period of three to four minutes. No purge flow indicates the trap is frozen. Simply leave the valve in the DESORB mode and heat the trap to 240°C for approximately five minutes. Do not turn the valve to the INJECT position for this will allow a massive amount of water to bombard the ion source and cause the instrument to go into high vacuum failure. If the trap freezes in one minute or less, put the trap through three heating cycles in the DESORB mode before attempting to analyze another cartridge.

18.2 Injection Procedure

18.2.1 At the end of the eight-minute desorption cycle, close the nitrogen toggle valve.

18.2.2 Turn the desorption unit valve to the INJECT mode (down).

18.2.3 Initiate heating of the nickel trap.

18.2.4 Strike a CARRIAGE RETURN key on the terminal to begin acquisition of data.

18.2.5 Turn off the trap after it has heated to 240°C.

18.2.6 Shut the GC oven door five minutes after injection has occurred.

18.2.7 To start the GC program, turn the switch on the far right of the GC controller to PROGRAM HOLD immediately after closing the door of the GC oven.

19.0 USE OF RESPONSE FACTORS IN THE QUANTITATION OF VOLATILE ORGANIC COMPOUNDS THERMALLY DESORBED FROM TENAX CARTRIDGES

Quantitation of volatile organic compounds is performed by the method of Response Factors. To determine a Response Factor, an analysis is performed of a cartridge containing known quantities of both the standard and the compound of interest. The Response Factor is calculated using the following equation:

RF = (peak area compound of interest/ng compound of interest)/(peak area standard/ng standard)

A set of two Tenax cartridges containing the standard and compounds of interest has been required in order to establish Response Factors for all of the compounds of interest for the TEAM study. The cartridges are designated as CPMX + RMR and RMR. The CPMX + RMR cartridges contain seven compounds used for determination of column performance parameters as well as target compounds which are loaded onto the cartridges via a Flash Evaporation System. RMR cartridges contain standard and other target compounds loaded via a Permeation System. A typical list of target compounds and number of nanograms/cartridge is presented below:

Compound	ng Loaded
Benzene	304
Chloroform	114
1,1,1-Trichloroethane	174
Carbon tetrachloride	201
1,2-Dichloroethane	329
Trichloroethylene	403
1,1,2,2-Tetrachloroethane	409
Chlorobenzene	140
Tetrachloroethylene	323
Ethylbenzene	346
p-Xylene	344
o-Xylene	352
Styrene	362
o-Dichlorobenzene	260
p-Dichlorobenzene	260
n-Octane	288
n-Decane	292
5-Nonanone*	328
Acetophenone*	411
2,6-Dimethylphenol*	294
2,6-Dimethylaniline*	391
1-Octanol*	330
Perfluorobenzene	125
Perfluorotoluene	130

*Used to calculate column performance parameters; not target compounds.

19.1 Establishment of a Response Factor Data Base

19.1.1 To establish a Response Factor data base for the target compounds prior to the analysis of field sample cartridges, analyze a series of five CPMX + RMR and five RMR cartridges by thermal desorption with GC/MS.

19.1.2 Calculate five sets of Response Factors for each ion of interest for each target compound.

19.1.3 Tabulate these Response Factors.

19.1.4 Calculate mean, standard deviation, and coefficient of variation.

19.1.5 If several Response Factors in a particular assay can be identified as outliers by their lack of correspondence to the other values obtained, discard that set of Response Factors.

19.1.6 Analyze another cartridge.

19.1.7 If the set of five Response Factors does not appear to be consistent, immediately check the desorption unit, gas chromatograph, and mass spectrometer for the presence of an air leak or some other problem and repeat the series of five cartridges.

19.2 Quantitation of Response Factors for Target Compounds

19.2.1 Initiate quantitation of Response Factors for target compounds by creating a Procedure in the Incos data system to obtain ion peak areas and scan numbers automatically for each ion of each compound.

NOTE: A Procedure is simply a string of commands concatenated to make data processing more efficient. The Procedure also incorporates a Method file which acts like a smaller Procedure.

An example of a Procedure named TEST for ions 117 and 186 of Perfluorobenzene and the 61, 97, and 99 ions of 1,1,1-Trichloroethane can be entered on the computer terminal as follows:

```
>TEST  =  SET Q $ <CR>
       =  EDQL (-; W; E) <CR>
       =  CHRO (I; R; 186; 117; X291; @BH; E) <CR>
       =  CHRO (I; R; 61; 97; 99; X334; @BH; E) <CR>
       =  QUAN (I; F1; H; E) <CR>
       =  FEED <CR>
       =  BEEP <CR>
       =  <CR>
```

19.2.2 Enter the Method file into the computer separately and designate the Method file by its own name.

NOTE: For example, Method file BH is entered in the following way:

```
>CHROBH = G-25,35;N-2,2;A>-2,2;D-25,45; <CR> <CR>
```

The Procedure includes information about compound ions, the scan number at which the compound should be sought, and the Method file. The Method file contains information needed to designate a mass range in which to search for ions of the target compounds and establishes parameters required for peak area quantitation. CPMX and RMR are the Procedure names currently being used for RMR and CPMX + RMR quantitation. A trace of each of the procedures is presented below:

TRACE OF PROCEDURE CPMX

```
* SETQ $
* EDQL (-;W;E)
* CHRO(I;R;186;117;X291;@BH;E)
* CHRO(I;R;61;97;99;X334;@BH;E)
* CHRO(I;R;117;119;X359;@BH;E)
* CHRO(I;R;83;85;X300;@BH;E)
* CHRO(I;R;62;64;X328;@BH;E)
* CHRO(I;R;78;X354;@BH;E)
* CHRO(I;R;95;130;132;X411;@BH;E)
* CHRO(I;R;164;166;X624;@BH;E)
* CHRO(I;R;77;112;114;X688;@BH;E)
* CHRO(I;R;83;166;168;X814;@BH;E)
* QUAN(I;F1;H;E)
* FEED
* BEEP
```

TRACE OF PROCEDURE RMR

```
* SETQ $
* EDQL (-;W;E)
* CHRO(I;R;186;117;X294;@BH;E)
* CHRO(I;R;104;X790;@BH;E)
* CHRO(I;R;146;148;X1067;@BH;E)
* CHRO(I;R;146;148;X1119;@BH;E)
* CHRO(I;R;91;106;X729;@BH;E)
* CHRO(I;R;91;106;X751;@BH;E)
* CHRO(I;R;91;106;X801;@BH;E)
* QUAN(I;F1;H;E)
* FEED
* BEEP
```

19.2.3 To use the CPMX Procedure after analyzing an RMR cartridge, simply type >CPMX <CR>.

NOTE: The computer automatically obtains quantitation data and prints a hardcopy of the quantitation date on the Versatec printer. An example of a quantitated RMR cartridge using Procedure CPMX is presented below:

```
QUANTITATION REPORT    FILE: 42224259

DATA: 42224259.TI
06/20/83 18:22:00
SAMPLE: INAIR 012-5 RMR-01
SUBMITTED BY: EP               ANALYST: RLP

AMOUNT=AREA(HGHT) * REF.AMNT/(REF.AREA(HGHT)* RESP.FACT)
RESP. FAC. FROM  LIBRARY ENTRY

 NO   M/E  SCAN   TIME  REF   RRT  METH    AREA(HGHT)  AMOUNT       %TOT
  1   117   311   5:11  30  0.385  A BB       72470.    9.070       2.46
  2   186   311   5:11  30  0.385  A BV      121673.   15.220       4.12
  3    61  *323   5:23  30  0.400  A BB         312.    0.039       0.01
  4    61  *340   5:40  30  0.421  A BV       19186.    2.401       0.65
  5    61   346   5:46  30  0.428  A VB       30855.    3.862       1.05
  6    97   346   5:46  30  0.428  A BB       78056.    9.769       2.64
  7    99   346   5:46  30  0.428  A BB       48061.    6.015       1.63
  8   117  *343   5:43  30  0.425  A BB       29007.    3.630       0.98
  9   117   371   6:11  30  0.455  A BB       55796.    6.983       1.89
 10   119  *346   5:46  30  0.428  A BB        8426.    1.055       0.29
 11   119   371   6:11  30  0.459  A BB       54174.    6.780       1.84
 12    83   317   5:17  30  0.392  A BB       94959.   11.885       3.22
 13    85   317   5:17  30  0.392  A BB       59753.    7.478       2.02
 14    62   340   5:40  30  0.421  A BE      172663.   21.610       5.85
 15    64   340   5:40  30  0.421  A BB       53752.    6.727       1.82
 16    78   366   6:06  30  0.453  A BB      373562.   46.753      12.66
 17    95   419   6:59  30  0.519  A BB      124031.   15.523       4.20
 18   130   418   6:58  30  0.517  A BB      149576.   18.720       5.07
 19   132   418   6:58  30  0.517  A BB      136260.   17.054       4.62
 20   164   612  10:12  30  0.757  A BV       81598.   10.212       2.76
 21   164 * 625  10:25  30  0.774  A VB        2587.    0.324       0.09
 22   166   612  10:12  30  0.757  A BV      100714.   12.605       3.41
 23   166 * 625  10:25  30  0.774  A VB        3475.    0.435       0.12
 24    77   677  11:17  30  0.838  A BV       43814.    5.483       1.49
 25    77 * 689  11:29  30  0.853  A VB        1681.    0.210       0.06
 26   112   677  11:17  30  0.838  A BV       83208.   10.414       2.82
 27   112 * 689  11:29  30  0.853  A VB        3335.    0.417       0.11
 28   114   677  11:17  30  0.838  A BV       26239.    3.284       0.[illegible]
 29   114 * 689  11:29  30  0.853  A VB        1340.    0.168       0.05
 30    83   808  13:28  30  1.000  A BB      799012.  100.000      27.07
 31   166   808  13:28  30  1.000  A BB       55008.    6.884       1.86
 32   166   808  13:28  30  1.000  A BB       67241.    8.416       2.28
```

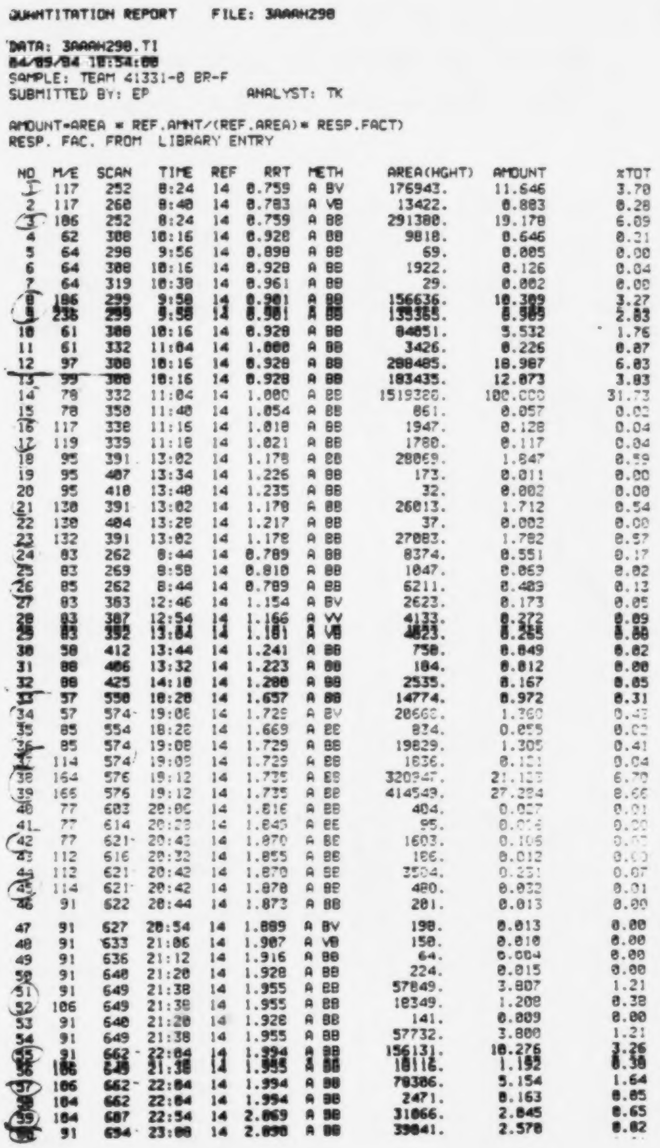
In the computer output presented above, some of the ions have asterisks beside them. These asterisks were inserted by the operator to indicate that some extraneous ions which do not belong to a target compound were quantitated by the computer. These extraneous ions were found in the same scan range and ion channel being monitored. All of these extraneous ions must be deleted to avoid reporting misleading and/or false data.

19.3 Deletion of Extraneous Ions from a Quantitation List

19.3.1 Use a program called the Quantitation List Editor (EDQL) to make deletions of extraneous ions.

19.3.2 Enter the EDQL program by typing >EDQL file name <CR>.

NOTE: A printout similar to that obtained from the QUAN program will be observed:



```
QUANTITATION REPORT    FILE: 3AAAH298

DATA: 3AAAH298.TI
04/09/94 10:54:00
SAMPLE: TEAM 41331-8 BR-F
SUBMITTED BY: EP                ANALYST: TK

AMOUNT=AREA * REF.AMNT/(REF.AREA)* RESP.FACT)
RESP. FAC. FROM  LIBRARY ENTRY

 NO  M/E  SCAN   TIME  REF   RRT   METH      AREA(HGHT)   AMOUNT       %TOT
  1  117   252   8:24   14  0.759  A BV       176943.     11.646       3.70
  2  117   260   8:40   14  0.783  A VB        13422.      0.883       0.28
  3  106   252   8:24   14  0.759  A BB       291380.     19.178       6.09
  4   62   308  10:16   14  0.928  A BB         9818.      0.646       0.21
  5   64   298   9:56   14  0.898  A BB           69.      0.005       0.00
  6   64   308  10:16   14  0.928  A BB         1922.      0.126       0.04
  7   64   319  10:38   14  0.961  A BB           29.      0.002       0.00
  8  106   299   9:58   14  0.901  A BB       156636.     10.309       3.27
  9  236   299   9:58   14  0.901  A BB       135365.      8.909       2.83
 10   61   308  10:16   14  0.928  A BB        84051.      5.532       1.76
 11   61   332  11:04   14  1.000  A BB         3426.      0.226       0.07
 12   97   308  10:16   14  0.928  A BB       288485.     18.987       6.03
 13   99   308  10:16   14  0.928  A BB       183435.     12.073       3.83
 14   78   332  11:04   14  1.000  A BB      1519320.    100.000      31.73
 15   78   350  11:40   14  1.054  A BB          861.      0.057       0.02
 16  117   338  11:16   14  1.018  A BB         1947.      0.128       0.04
 17  119   339  11:18   14  1.021  A BB         1780.      0.117       0.04
 18   95   391  13:02   14  1.178  A BB        28069.      1.847       0.59
 19   95   407  13:34   14  1.226  A BB          173.      0.011       0.00
 20   95   410  13:40   14  1.235  A BB           32.      0.002       0.00
 21  130   391  13:02   14  1.178  A BB        26013.      1.712       0.54
 22  130   404  13:28   14  1.217  A BB           37.      0.002       0.00
 23  132   391  13:02   14  1.178  A BB        27083.      1.782       0.57
 24   83   262   8:44   14  0.789  A BB         8374.      0.551       0.17
 25   83   269   8:58   14  0.810  A BB         1047.      0.069       0.02
 26   85   262   8:44   14  0.789  A BB         6211.      0.409       0.13
 27   83   383  12:46   14  1.154  A BV         2623.      0.173       0.05
 28   83   387  12:54   14  1.166  A VV         4133.      0.272       0.09
 29   83   392  13:04   14  1.181  A VB         4023.      0.265       0.08
 30   58   412  13:44   14  1.241  A BB          750.      0.049       0.02
 31   88   406  13:32   14  1.223  A BB          184.      0.012       0.00
 32   88   425  14:10   14  1.280  A BB         2535.      0.167       0.05
 33   57   550  18:20   14  1.657  A BB        14774.      0.972       0.31
 34   57   574  19:08   14  1.729  A BV        20660.      1.360       0.43
 35   85   554  18:28   14  1.669  A BB          834.      0.055       0.02
 36   85   574  19:08   14  1.729  A BB        19829.      1.305       0.41
 37  114   574  19:08   14  1.729  A BB         1836.      0.121       0.04
 38  164   576  19:12   14  1.735  A BB       320947.     21.123       6.70
 39  165   576  19:12   14  1.735  A BB       414549.     27.284       8.66
 40   77   603  20:06   14  1.816  A BB          404.      0.027       0.01
 41   77   614  20:28   14  1.849  A BB           95.      0.006       0.00
 42   77   621  20:42   14  1.870  A BB         1603.      0.106       0.03
 43  112   616  20:32   14  1.855  A BB          186.      0.012       0.00
 44  112   621  20:42   14  1.870  A BB         3504.      0.231       0.07
 45  114   621  20:42   14  1.870  A BB          480.      0.032       0.01
 46   91   622  20:44   14  1.873  A BB          201.      0.013       0.00

 47   91   627  20:54   14  1.889  A BV          198.      0.013       0.00
 48   91   633  21:06   14  1.907  A VB          150.      0.010       0.00
 49   91   636  21:12   14  1.916  A BB           64.      0.004       0.00
 50   91   640  21:20   14  1.928  A BB          224.      0.015       0.00
 51   91   649  21:38   14  1.955  A BB        57849.      3.807       1.21
 52  106   649  21:38   14  1.955  A BB        18349.      1.208       0.38
 53   91   640  21:20   14  1.928  A BB          141.      0.009       0.00
 54   91   649  21:38   14  1.955  A BB        57732.      3.800       1.21
 55   91   662  22:04   14  1.994  A BB       156131.     10.276       3.26
 56  106   649  21:38   14  1.955  A BB        18116.      1.192       0.38
 57  106   662  22:04   14  1.994  A BB        78306.      5.154       1.64
 58  104   662  22:04   14  1.994  A BB         2471.      0.163       0.05
 59  104   687  22:54   14  2.069  A BB        31066.      2.045       0.65
 60   91   694  23:08   14  2.090  A BB        39041.      2.570       0.82
```

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19.3.3 Make all deletions from the bottom to the top of the list in EDQL to avoid having entry numbers changed by the computer and thus making incorrect deletions.

19.3.4 From inspection of the Quantitation Report, one sees asterisks at numbers 28, 27, 25, 23, 21, 10, 8, 4 and 3.

19.3.5 In the EDQL program, type EDQL: -28; -27; -25; -23; -21; -10; -8; -4; -3 <CR> and a revised quantitation list with no extraneous ions will appear.

19.3.6 Type EDQL: W <CR> to save all of these data and write the data to an output file for future use.

NOTE: The updated EDQL list is presented below:

```
REFERENCE RETENTION TIME (MIN:SEC)    11:04
 NO. LOW MASS   HIGH   SCAN    TIME  METH        AREA    HEIGHT   NAME NUM
   1  116.54  117.54    252    8:24  A BV     176943.    38830.   --     0
   2  185.56  186.56    252    8:24  A BB     291380.    57274.   --     0
   3  185.56  186.56    299    9:58  A BB     156636.    31542.   --     0
   4  235.57  236.57    299    9:58  A BB     135365.    26841.   --     0
   5   60.52   61.52    308   10:16  A BB      84051.    18887.   --     0
   6   96.53   97.53    308   10:16  A BB     288485.    66035.   --     0
   7   98.53   99.53    308   10:16  A BB     183435.    41737.   --     0
   8   77.52   78.52    332   11:04  A BB    1519380.   246528.   --     0
   9  116.54  117.54    338   11:16  A BB       1947.      514.   --     0
  10  118.54  119.54    339   11:18  A BB       1780.      533.   --     0
  11   94.53   95.53    391   13:02  A BB      28069.     5437.   --     0
  12  129.54  130.54    391   13:02  A BB      26013.     5409.   --     0
  13  131.54  132.54    391   13:02  A BB      27083.     5488.   --     0
  14   82.53   83.53    262    8:44  A BB       8374.     1860.   --     0
  15   84.53   85.53    262    8:44  A BB       6211.     1178.   --     0
  16   56.52   57.52    574   19:08  A BV      20668.     5638.   --     0
  17   84.53   85.53    574   19:08  A BB      19829.     7360.   --     0
  18  113.53  114.53    574   19:08  A BB       1836.      866.   --     0
  19  163.55  164.55    576   19:12  A BB     320947.   121344.   --     0
  20  165.55  166.55    576   19:12  A BB     414549.   156928.   --     0
  21   76.52   77.52    621   20:42  A BB       1603.      576.   --     0
  22  113.53  114.53    621   20:42  A BB        480.      222.   --     0
  23   90.53   91.53    649   21:38  A BB      57849.    26488.   --     0
  24  105.53  106.53    649   21:38  A BB      18349.     8448.   --     0
  25   90.53   91.53    662   22:04  A BB     156131.    50875.   --     0
  26  105.53  106.53    662   22:04  A BB      78306.    25852.   --     0
  27  103.53  104.53    687   22:54  A BB      31066.    14144.   --     0
  28   90.53   91.53    694   23:08  A BB      39041.    15607.   --     0
  29  105.53  106.53    694   23:08  A BB      18565.     7848.   --     0
  30  145.54  146.54    847   28:14  A BB      24551.    12112.   --     0
  31  147.54  148.54    847   28:14  A BB      16635.     7872.   --     0
  32  141.54  142.54    851   28:22  A BB       1139.      781.   --     0
  33  145.54  146.54    876   29:12  A BB       3734.     2092.   --     0
  34  147.54  148.54    876   29:12  A BB       2425.     1310.   --     0
  35  120.54  121.54    768   25:36  A BB       1324.      851.   --     0
  36  135.54  136.54    768   25:36  A BB        748.      492.   --     0
  37  155.55  156.55    970   32:20  A BB        844.      683.   --     0
  38   84.53   85.53   1079   35:58  A BB       4357.     2308.   --     0
  39  169.55  170.55   1078   35:56  A BB        216.      113.   --     0
```

19.4 Creation of Libraries for CPMX + RMR and RMR Compounds

The next step in calculation of Response Factors is creation of appropriate libraries for the target compounds.

19.4.1 Designate each library by a two-letter name.

19.4.2 Do not attempt to create more than one library at a time.

19.4.3 Before entry of a specific compound in the library, list the library name under SET4 of the STAT program.

NOTE: The library in current use for the RMR target compounds is designated library SR.

19.4.4 To enter library name SR in SET4 under the STAT program type >STAT <CR> followed by >SET4 SR <CR>.

19.4.5 Get a mass spectrum for each compound in library SR.

19.4.6 Obtain a scan number for each compound from the Quantitation List for an RMR cartridge analysis.

19.4.7 Use the scan number for a compound under the CHRO program for a library search.

19.4.8 Enter CHRO by typing >CHRO file name, cali table <CR>.

19.4.9 Be sure that the standard compound ion to be used as the quantitation standard is the first entry in a library.

NOTE: Since the ion of mass 186 of perfluorobenzene is this ion, make it the first entry of library SR.

19.4.10 If Scan #260 corresponds to the elution position of perfluorobenzene (PFB), type CHRO: S260;Y <CR>.

19.4.11 To enter the library editor program (EDLB) from the library search type LIBR: ED <CR>.

19.4.12 Type EDLB: I <CR> and answer the list of questions which follows:

NEW NAME: Type Perfluorobenzene <CR>
NEW FORMULA: Type C6 F6 <CR>
MOLECULAR WEIGHT: Type 186 <CR>
RETENTION TIME: Type <CR>
BASE PEAK INTENSITY: Type <CR>
USER PARAMETER # 1: Type <CR>
USER PARAMETER # 2: Type <CR>

RELATIVE RETENTION TIME: Type 1.000 <CR>

QUANTITATION MASS: Type 186 <CR>

STANDARD AMOUNT: Type 125 <CR>

UNITS: Type NG/CT <CR>

REFERENCE PEAK (NAME,NUM): Type SR,1 <CR>

RESPONSE FACTOR: Type <CR>

19.4.13 Disregard questions Retention Time and Base Peak Intensity because these questions are answered by the computer.

19.4.14 Disregard User Parameter #1 and #2 and Response Factor as these questions are not applicable at this point.

19.4.15 Calculate the Relative Retention Time by dividing the scan number of the target compound into the scan number of the standard (PFB).

19.4.16 Remember, the first entry of a quantitation library must be the ion of the standard compound against which quantitation is to be performed, so make the 186 ion of PFT the first entry.

19.4.17 Obtain the value for Standard Amount from the person who has loaded the cartridges.

NOTE: In library SR the Reference Peak and Number will always be answered SR,1 so that a library entry and an ion of a particular target compound can be correlated and quantitated against the standard ion.

19.4.18 After the questions have been answered, verify the correctness of all the information entered for the ion of a compound by inspecting the terminal display to be entered in library SR.

19.4.19 If no errors have been made in the entry of the information, simply strike <CR> to enter the EDLB program.

19.4.20 If there is more than one ion which needs to be entered for a given compound, type EDLB: I' <CR> again.

19.4.21 Answer all the questions in the same way except for the Quantitation Mass which must be changed to the other ion from the compound of interest.

19.4.22 If no change is necessary for a given answer, strike <CR>.

19.4.23 Should an error be discovered at the end of the list of questions, type N <CR> to enter EDLB.

19.4.24 Then type EDLB: I' <CR> to reenter the correct responses.

19.4.25 When all the ions desired for quantitation for a given compound have been entered into the library, return to the CHRO program by typing EDLB: E <CR> and LIBR: E <CR>.

19.4.26 Find the scan number for the next target compound and repeat the above steps for entry into the library until all the ions of all the compounds have been entered.

19.4.27 Obtain a list of all the library entries in library SR by typing >SET6 SR.LL <CR>.

19.4.28 Type >EDLL <CR>, followed by EDLL: 1,n where n is the total number of ions entered in library RR.

19.4.29 Type EDLL: B <CR> and a printout of library SR will appear at the Versatec printer.

19.4.30 Repeat all of the above steps for the compounds on the CPMX + RMR cartridges.

19.5 Correlation of Library Entry and Quantitation List

19.5.1 When both a Quantitation List and a library have been created, obtain calculated Response Factors by making a correlation between the ion of the compound and a library entry number.

19.5.2 Enter EDQL again by typing >EDQL file name <CR>.

19.5.3 Using library SR and a Quantitation List for an RMR cartridge type EDQL: C library SR <CR>.

NOTE: The computer then asks for the library name and the entry number that correlate to a compound ion. For example, when the 186 ion of PFB is listed type EDQL: SR,1 <CR> to correlate the first entry in library SR with the 186 ion of PFB.

19.5.4 Correlate all the ions to a library entry as they appear under EDQL.

NOTE: When all ions have been correlated, EDQL will display a summary table containing ions, scan number, peak area, library name, and entry number.

19.5.5 Verify that no errors have been made, then type EDQL: W <CR> to write the data back to the Quantitation List.

19.5.6 Exit from EDQL by typing EDQL: E <CR> and type > QUAN file name <CR>.

19.5.7 Under QUAN type QUAN: F2;H <CR> and a hardcopy of Response Factors for the various ions will be printed on the Versatec printer (see Figure 16).

19.5.8 Store these Response Factors in a Response List of the computer by typing QUAN: > <CR>.

19.5.9 Repeat these same steps for the CPMX + RMR quantitation list and the appropriate library.

19.6 Sample Quantitation

19.6.1 Write a procedure to allow quantitation of all desired ions of of all target compounds to perform sample quantitation.

19.6.2 Create a library of all target compounds.

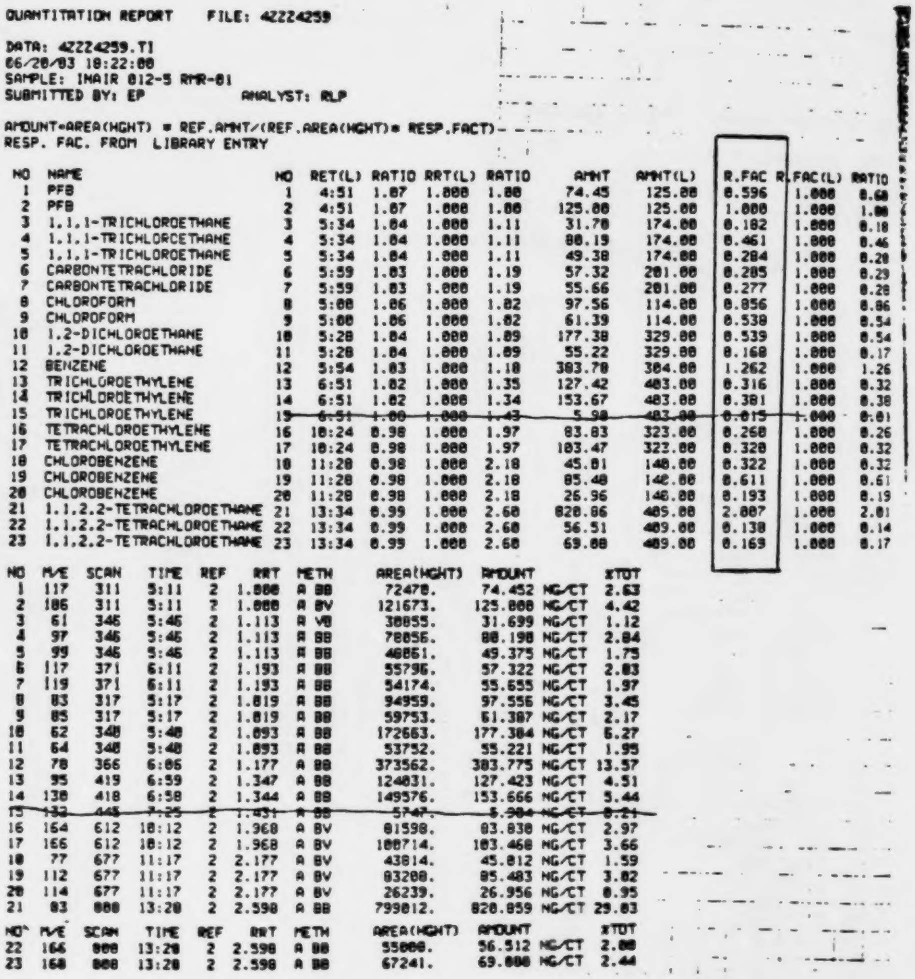
19.6.3 Use the same procedure as the Procedure and library for the RMR and CPMX + RMR compounds, with two exceptions: when the compound ion is entered into the library and the computer asks a series of questions, the mean Response Factor calculated from the five initial analyses of CPMX + RMR cartridges is entered when the computer requests a Response Factor.

19.6.4 Be sure to list a mean Response Factor for all the compound ions in the sample quantitation library.

19.6.5 When a printout under QUAN of the final numbers is desired, delete the extraneous ions as before, type EDQL: W <CR>, and then exit by typing EDQL: E <CR>.

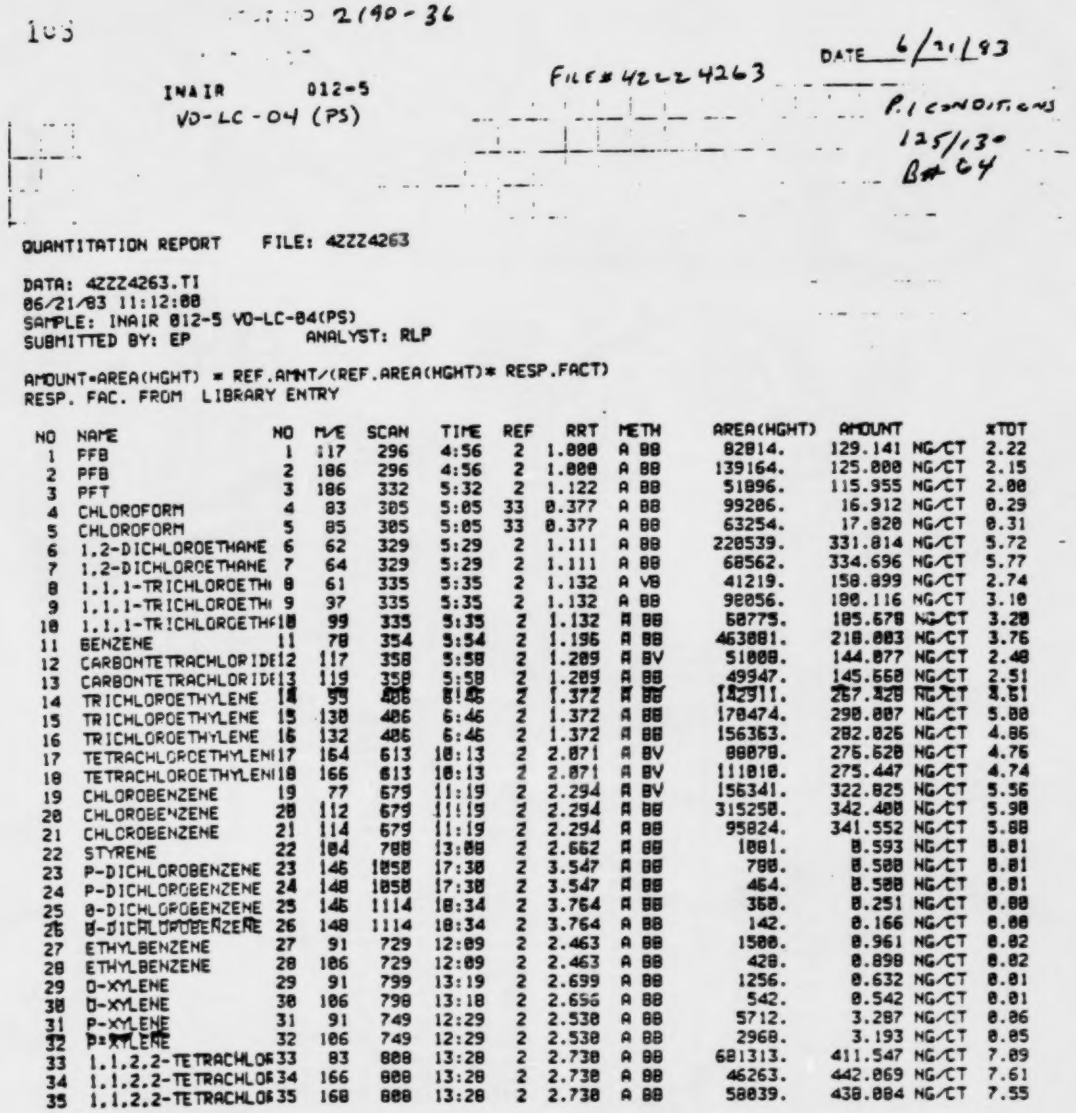
19.6.6 To obtain the final answer in ng/cart, go into the QUAN program.

19.6.7 Type >QUAN filename <CR> followed by QUAN: H <CR> (not QUAN: F2;H as before) (see Figure 17 for an example).



```
QUANTITATION REPORT    FILE: 42224259

DATA: 42224259.TI
06/20/83 10:22:00
SAMPLE: INAIR 012-5 RMR-01
SUBMITTED BY: EP              ANALYST: RLP

AMOUNT=AREA(HGHT) * REF.AMNT/(REF.AREA(HGHT)* RESP.FACT)
RESP. FAC. FROM  LIBRARY ENTRY

 NO  NAME                          NO  RET(L) RATIO RRT(L) RATIO    AMNT  AMNT(L)   R.FAC R.FAC(L) RATIO
  1  PFB                            1   4:51  1.07  1.000  1.00    74.45   125.00   0.596   1.000   0.60
  2  PFB                            2   4:51  1.07  1.000  1.00   125.00   125.00   1.000   1.000   1.00
  3  1.1.1-TRICHLOROETHANE          3   5:34  1.04  1.000  1.11    31.70   174.00   0.182   1.000   0.18
  4  1.1.1-TRICHLORCETHANE          4   5:34  1.04  1.000  1.11    80.19   174.00   0.461   1.000   0.46
  5  1.1.1-TRICHLOROETHANE          5   5:34  1.04  1.000  1.11    49.38   174.00   0.284   1.000   0.28
  6  CARBONTETRACHLORIDE            6   5:59  1.03  1.000  1.19    57.32   201.00   0.285   1.000   0.29
  7  CARBONTETRACHLORIDE            7   5:59  1.03  1.000  1.19    55.66   201.00   0.277   1.000   0.28
  8  CHLOROFORM                     8   5:00  1.06  1.000  1.02    97.56   114.00   0.856   1.000   0.86
  9  CHLOROFORM                     9   5:00  1.06  1.000  1.02    61.39   114.00   0.538   1.000   0.54
 10  1.2-DICHLOROETHANE            10   5:20  1.04  1.000  1.09   177.38   329.00   0.539   1.000   0.54
 11  1.2-DICHLOROETHANE            11   5:20  1.04  1.000  1.09    55.22   329.00   0.168   1.000   0.17
 12  BENZENE                       12   5:54  1.03  1.000  1.10   303.78   304.00   1.262   1.000   1.26
 13  TRICHLOROETHYLENE             13   6:51  1.02  1.000  1.35   127.42   403.00   0.316   1.000   0.32
 14  TRICHLOROETHYLENE             14   6:51  1.02  1.000  1.34   153.67   403.00   0.381   1.000   0.38
 15  TRICHLOROETHYLENE         ~~  15   6:51  1.00  1.000  1.43     5.90   403.00   0.015   1.000   0.01~~
 16  TETRACHLOROETHYLENE           16  10:24  0.98  1.000  1.97    83.83   323.00   0.260   1.000   0.26
 17  TETRACHLOROETHYLENE           17  10:24  0.98  1.000  1.97   103.47   322.00   0.320   1.000   0.32
 18  CHLOROBENZENE                 18  11:20  0.98  1.000  2.18    45.01   140.00   0.322   1.000   0.32
 19  CHLOROBENZENE                 19  11:20  0.98  1.000  2.18    85.48   140.00   0.611   1.000   0.61
 20  CHLOROBENZENE                 20  11:20  0.98  1.000  2.18    26.96   140.00   0.193   1.000   0.19
 21  1.1.2.2-TETRACHLOROETHANE     21  13:34  0.99  1.000  2.60   820.86   409.00   2.007   1.000   2.01
 22  1.1.2.2-TETRACHLOROETHANE     22  13:34  0.99  1.000  2.60    56.51   409.00   0.138   1.000   0.14
 23  1.1.2.2-TETRACHLOROETHANE     23  13:34  0.95  1.000  2.60    69.00   409.00   0.169   1.000   0.17

 NO  M/E  SCAN   TIME  REF   RRT  METH   AREA(HGHT)   AMOUNT         %TOT
  1  117   311   5:11   2  1.000  A BB     72470.     74.452 NG/CT   2.63
  2  106   311   5:11   ?  1.000  A BV    121673.    125.000 NG/CT   4.42
  3   61   346   5:46   2  1.113  A VB     30055.     31.699 NG/CT   1.12
  4   97   346   5:46   2  1.113  A BB     78056.     80.198 NG/CT   2.84
  5   99   346   5:46   2  1.113  A BB     48061.     49.375 NG/CT   1.75
  6  117   371   6:11   2  1.193  A BB     55796.     57.322 NG/CT   2.03
  7  119   371   6:11   2  1.193  A BB     54174.     55.655 NG/CT   1.97
  8   83   317   5:17   2  1.019  A BB     94959.     97.556 NG/CT   3.45
  9   85   317   5:17   2  1.019  A BB     59753.     61.387 NG/CT   2.17
 10   62   340   5:40   2  1.093  A BB    172663.    177.384 NG/CT   6.27
 11   64   340   5:40   2  1.093  A BB     53752.     55.221 NG/CT   1.95
 12   78   366   6:06   2  1.177  A BB    373562.    303.775 NG/CT  13.57
 13   95   419   6:59   2  1.347  A BB    124031.    127.423 NG/CT   4.51
 14  130   418   6:58   2  1.344  A BB    149576.    153.666 NG/CT   5.44
~~15  132   445   7:25   2  1.431  A BB      5747.      5.904 NG/CT   0.21~~
 16  164   612  10:12   2  1.968  A BV     81598.     83.830 NG/CT   2.97
 17  166   612  10:12   2  1.968  A BV    100714.    103.468 NG/CT   3.66
 18   77   677  11:17   2  2.177  A BV     43814.     45.012 NG/CT   1.59
 19  112   677  11:17   2  2.177  A BV     83208.     85.483 NG/CT   3.02
 20  114   677  11:17   2  2.177  A BV     26239.     26.956 NG/CT   0.95
 21   83   000  13:20   2  2.598  A BB    799012.    820.859 NG/CT  29.03
 NO  M/E  SCAN   TIME  REF   RRT  METH   AREA(HGHT)   AMOUNT         %TOT
 22  166   000  13:20   2  2.598  A BB     55000.     56.512 NG/CT   2.00
 23  168   000  13:20   2  2.598  A BB     67241.     69.000 NG/CT   2.44
```

Figure 16. Quantitation report for a RMR run showing response factor.



105

2190-36

FILE# 42ZZ4263

DATE 6/21/83

INAIR 012-5
VO-LC-04 (PS)

P.I CONDITIONS
125/130
B# 64

```
QUANTITATION REPORT   FILE: 42ZZ4263

DATA: 42ZZ4263.TI
06/21/83 11:12:00
SAMPLE: INAIR 012-5 VO-LC-04(PS)
SUBMITTED BY: EP             ANALYST: RLP

AMOUNT=AREA(HGHT) * REF.AMNT/(REF.AREA(HGHT)* RESP.FACT)
RESP. FAC. FROM  LIBRARY ENTRY

 NO  NAME                NO  M/E  SCAN  TIME  REF   RRT  METH     AREA(HGHT)  AMOUNT           %TOT
  1  PFB                  1  117   296  4:56   2  1.000  A BB        82814.   129.141 NG/CT   2.22
  2  PFB                  2  186   296  4:56   2  1.000  A BB       139164.   125.000 NG/CT   2.15
  3  PFT                  3  186   332  5:32   2  1.122  A BB        51896.   115.955 NG/CT   2.00
  4  CHLOROFORM           4   83   305  5:05  33  0.377  A BB        99206.    16.912 NG/CT   0.29
  5  CHLOROFORM           5   85   305  5:05  33  0.377  A BB        63254.    17.820 NG/CT   0.31
  6  1.2-DICHLOROETHANE   6   62   329  5:29   2  1.111  A BB       220539.   331.814 NG/CT   5.72
  7  1.2-DICHLOROETHANE   7   64   329  5:29   2  1.111  A BB        68562.   334.696 NG/CT   5.77
  8  1.1.1-TRICHLOROETH   8   61   335  5:35   2  1.132  A VB        41219.   158.899 NG/CT   2.74
  9  1.1.1-TRICHLOROETH   9   97   335  5:35   2  1.132  A BB        98056.   180.116 NG/CT   3.10
 10  1.1.1-TRICHLOROETH  10   99   335  5:35   2  1.132  A BB        60775.   185.678 NG/CT   3.20
 11  BENZENE             11   78   354  5:54   2  1.196  A BB       463001.   218.003 NG/CT   3.76
 12  CARBONTETRACHLORIDE12  117   358  5:58   2  1.209  A BV        51008.   144.077 NG/CT   2.48
 13  CARBONTETRACHLORIDE13  119   358  5:58   2  1.209  A BB        49947.   145.660 NG/CT   2.51
 14  TRICHLOROETHYLENE   14   95   406  6:46   2  1.372  A BB       142911.   267.420 NG/CT   4.61
 15  TRICHLOROETHYLENE   15  130   406  6:46   2  1.372  A BB       170474.   290.007 NG/CT   5.00
 16  TRICHLOROETHYLENE   16  132   406  6:46   2  1.372  A BB       156363.   282.026 NG/CT   4.86
 17  TETRACHLOROETHYLEN  17  164   613 10:13   2  2.071  A BV        80070.   276.620 NG/CT   4.76
 18  TETRACHLOROETHYLEN  18  166   613 10:13   2  2.071  A BV       111010.   275.447 NG/CT   4.74
 19  CHLOROBENZENE       19   77   679 11:19   2  2.294  A BV       156341.   322.825 NG/CT   5.56
 20  CHLOROBENZENE       20  112   679 11:19   2  2.294  A BB       315250.   342.400 NG/CT   5.90
 21  CHLOROBENZENE       21  114   679 11:19   2  2.294  A BB        95024.   341.552 NG/CT   5.88
 22  STYRENE             22  104   788 13:08   2  2.662  A BB         1001.     0.593 NG/CT   0.01
 23  P-DICHLOROBENZENE   23  146  1050 17:30   2  3.547  A BB          700.     0.500 NG/CT   0.01
 24  P-DICHLOROBENZENE   24  148  1050 17:30   2  3.547  A BB          464.     0.500 NG/CT   0.01
 25  O-DICHLOROBENZENE   25  146  1114 18:34   2  3.764  A BB          360.     0.251 NG/CT   0.00
 26  O-DICHLOROBENZENE   26  148  1114 18:34   2  3.764  A BB          142.     0.166 NG/CT   0.00
 27  ETHYLBENZENE        27   91   729 12:09   2  2.463  A BB         1500.     0.961 NG/CT   0.02
 28  ETHYLBENZENE        28  106   729 12:09   2  2.463  A BB          420.     0.898 NG/CT   0.02
 29  O-XYLENE            29   91   799 13:19   2  2.699  A BB         1256.     0.632 NG/CT   0.01
 30  O-XYLENE            30  106   798 13:18   2  2.656  A BB          542.     0.542 NG/CT   0.01
 31  P-XYLENE            31   91   749 12:29   2  2.530  A BB         5712.     3.287 NG/CT   0.06
 32  P-XYLENE            32  106   749 12:29   2  2.530  A BB         2960.     3.193 NG/CT   0.05
 33  1.1.2.2-TETRACHLOR  33   83   808 13:28   2  2.730  A BB       681313.   411.547 NG/CT   7.09
 34  1.1.2.2-TETRACHLOR  34  166   808 13:28   2  2.730  A BB        46263.   442.069 NG/CT   7.61
 35  1.1.2.2-TETRACHLOR  35  168   808 13:28   2  2.730  A BB        50039.   438.004 NG/CT   7.55
```

Figure 17. Completed sample quantitation report.

20.0 SEQUENCE OF SAMPLES

20.1 On each day that analysis is performed, analyze a CPMX + RMR cartridge first, then an RMR cartridge.

20.2 Perform all Quality Control calculations before any more analysis is initiated.

20.3 If the Quality Control calculations, PFT tune, and Response Factors all meet specifications, analyze a blank Tenax sample (either a laboratory blank or a field blank).

20.4 Following the analysis of the blank sample, analyze the remaining cartridges in no particular order, but analyze a control sample daily if one is available.

20.5 At the conclusion of each day of analysis, analyze either a CPMX + RMR or RMR cartridge, so that a complete set of three CPMX + RMR and RMR cartridges is analyzed every two days.

21.0 STORAGE OF DATA

21.1 Transfer all data acquired on the Finnigan 3300 to nine-track magnetic tapes for archiving and possible further reference.

21.2 If possible, avoid transfer of data to magnetic tape until all processing has been completed.

NOTE: Two complete disk images can be stored on a single 1200' magnetic tape.

21.3 Take two data disks where all data are processed and put one in unit #0 of the top disk drive and the other in unit #2 of the bottom disk drive.

21.4 Install a magnetic tape on the tape drive by referring to the diagram located in the lower right corner of the tape drive.

21.5 Depress the power switches at the top and bottom of the tape drive.

21.6 Depress the LOAD switch.

NOTE: At this point the POWER, LOAD, and ON LINE switches will be lit.

NOTE: All magnetic taping must be done from the master terminal which is the terminal used by the FINN 4021 operator.

21.7 On the master terminal type > ID <CR> and a + will appear on the screen.

21.8 Type + DTDW <CR> followed by H (no CR).

NOTE: A list of choices describing the capabilities of this program will appear.

21.9 Transfer one disk image at a time to tape.

NOTE: No carriage returns are necessary in the DTDW program.

21.10 Tell the computer initially what disk unit is to be copied by typing + U followed by the number of the disk to be taped.

21.11 Type + D and the first disk will be copied to the magnetic tape.

NOTE: This is considered the first file on the tape.

21.12 For the second disk type + U and disk number also.

21.13 Type + D again.

21.14 When both disk images are copied depress RESET and REWIND buttons on the tape drive to rewind the tape back to the LOAD position.

21.15 Depress the ON LINE switch.

21.16 Verify the disk images to ensure no error has been made in copying to the magnetic tape.

21.17 Type + U and the disk number copied on the first file.

21.18 Type + V.

NOTE: The same procedure is followed for the second disk image.

21.19 When both images are verified type + B to bootstrap the computer back to the MSDS operating system.

21.20 Remove the magnetic tape from the tape deck.

21.21 Replace the tape in the tape seal.

21.22 Label the tape with the next number for the sequence for Finnigan 3300 tapes, either RP-XYZ,1 for the first tape file or RP-XYZ,2 for the second tape file.

21.23 Make the appropriate entry in the tape log.

21.24 Make the appropriate entry in the instrument log so that a data file is readily correlated with a magnetic tape and one of the two files on that tape.

22.0 QUALITY CONTROL

Quality Control for the Finnigan 3300 requires calculations on peaks from the CPMX + RMR cartridge, the % RA of major ions of PFT, and response factors of the target compounds in the CPMX + RMR and RMR samples. It is extremely important these criteria be met before running any samples to ensure quality data.

22.1 Peak Asymmetry Calculations

22.1.1 Utilize a column performance Tenax cartridge to assure that the GM/MS/DS is operating properly.

22.1.2 Check the instrument tune stability as well as capillary column performance.

22.1.3 Calculate a peak asymmetry to measure peak tailing for the compounds acetophenone, 5-nonanone, and 1-octanol.

NOTE: The formula is % PAF = F/B x 100 where B equals the width of the back half of the peak 10% above the baseline and F equals the width of the front half of the peak 10% above the baseline (see Figure 18). The specifications acceptable for these compounds are as follows:

Acetophenone < 300
5-Nonanone < 160
1-Octanol < 250

22.2 Separation Number

22.2.1 Calculate the separation number for octane and decane to measure the separation efficiency of the capillary column.

NOTE: A value > 40 is acceptable. The formula is as follows:

$$SN = \left(\frac{D}{W_1 + W_2}\right) - 1$$

where D equals the distance between the two peaks on the RIC whereas W_1 and W_2 equal the width of the peak at 1/2 height (see Figure 19).

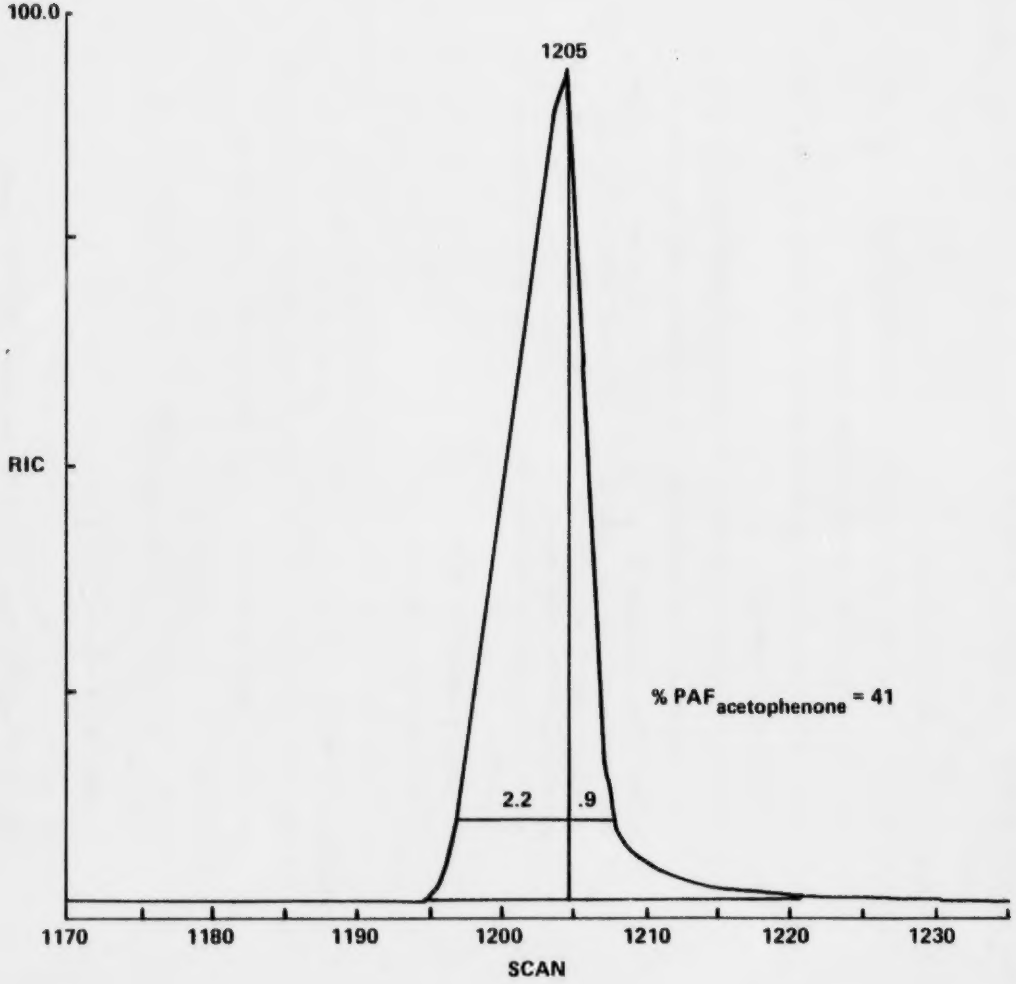


Figure 18. Peak asymmetry for acetophenone.

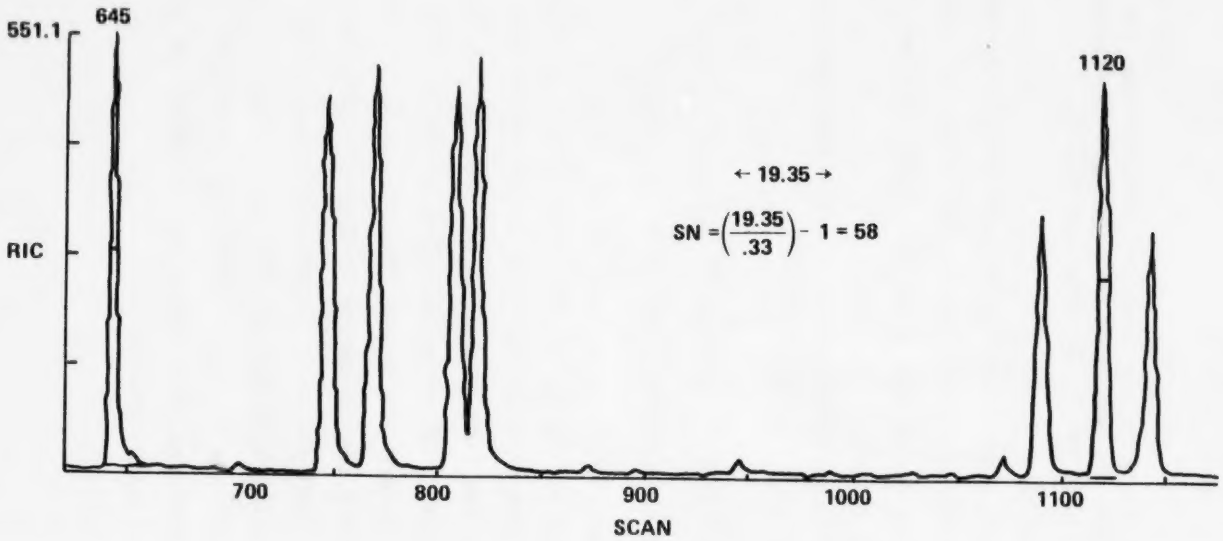


Figure 19. Separation number between octane and decane.

22.3 Resolution

22.3.1 Calculate a resolution by measuring ethylbenzene and p-xylene peaks in the RIC.

NOTE: The formula is as follows:

$$R = \frac{2D}{W_1+W_2}$$

where D equals the distance between the two peaks and W_1, W_2 equal the width of each peak on the baseline. This value must be > 1.0 (see Figure 20).

22.4 Acid/Base Ratios

22.4.1 To obtain the last column performance parameters, calculate an acid/base ratio between RIC areas of 2,6-dimethylaniline (DMA) and 2,6-dimethylphneol (DMP) versus RIC area of acetophenone.

NOTE: The formulas for acidity and basicity are as follows:

$$\text{Acidity} = \frac{\text{RIC area DMA}}{\text{RIC area acetophenone}}$$

$$\text{Basicity} = \frac{\text{RIC area DMP}}{\text{RIC area acetophenone}}$$

Acceptable values range from 0.7 to 1.3.

22.5 Mass Spectrum of PFT

22.5.1 Obtain a listing of PFT as well as a plotted mass spectrum showing the % RA of the major ions.

22.5.2 Check the tune stability by comparing to % RA of the Finnigan 3300 as established historically.

22.6 Computer Procedure for Column Performance Calculations

All data needed for column performance calculations can be obtained through the CHRO program. A procedure named CPMX has been written to automatically hardcopy all RIC chromatograms needed. To utilize this procedure simply type > CPMX <CR> after the CPMX + RMR sample has been run (see Figure 21) or generate appropriate hardcopy by individual instructions.

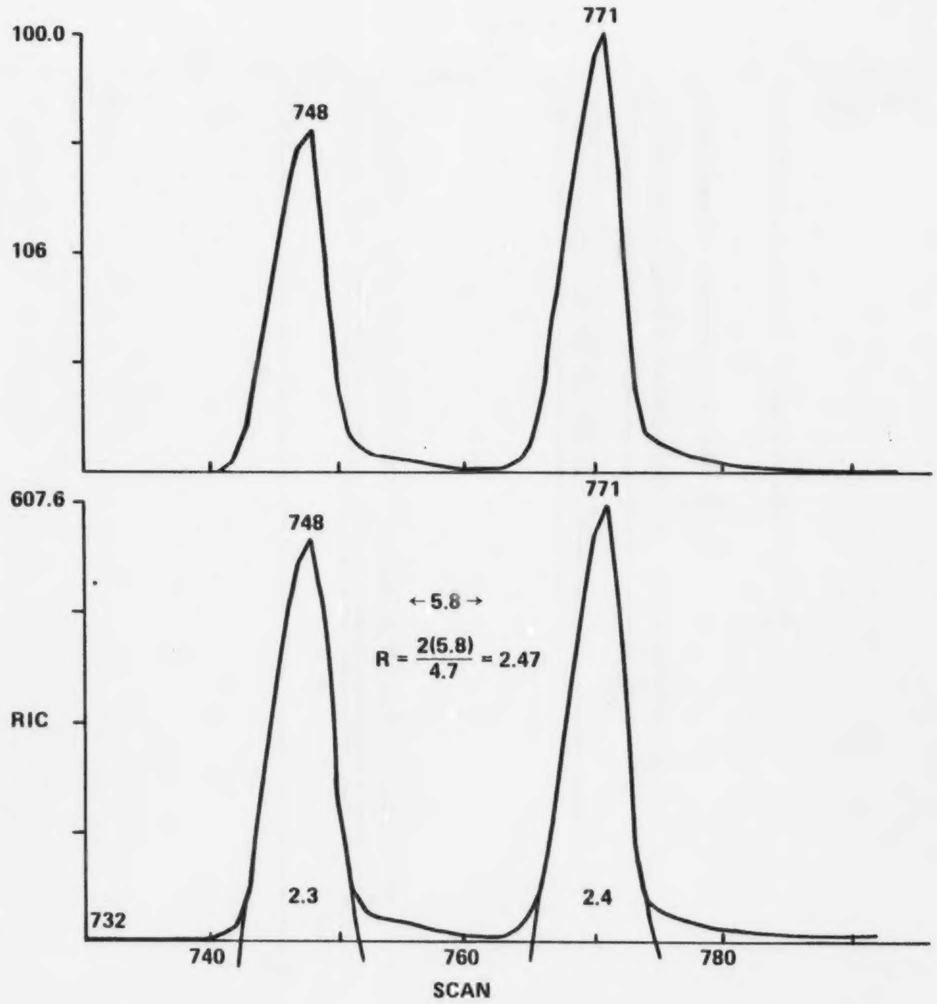


Figure 20. Resolution between ethylbenzene and p-xylene.

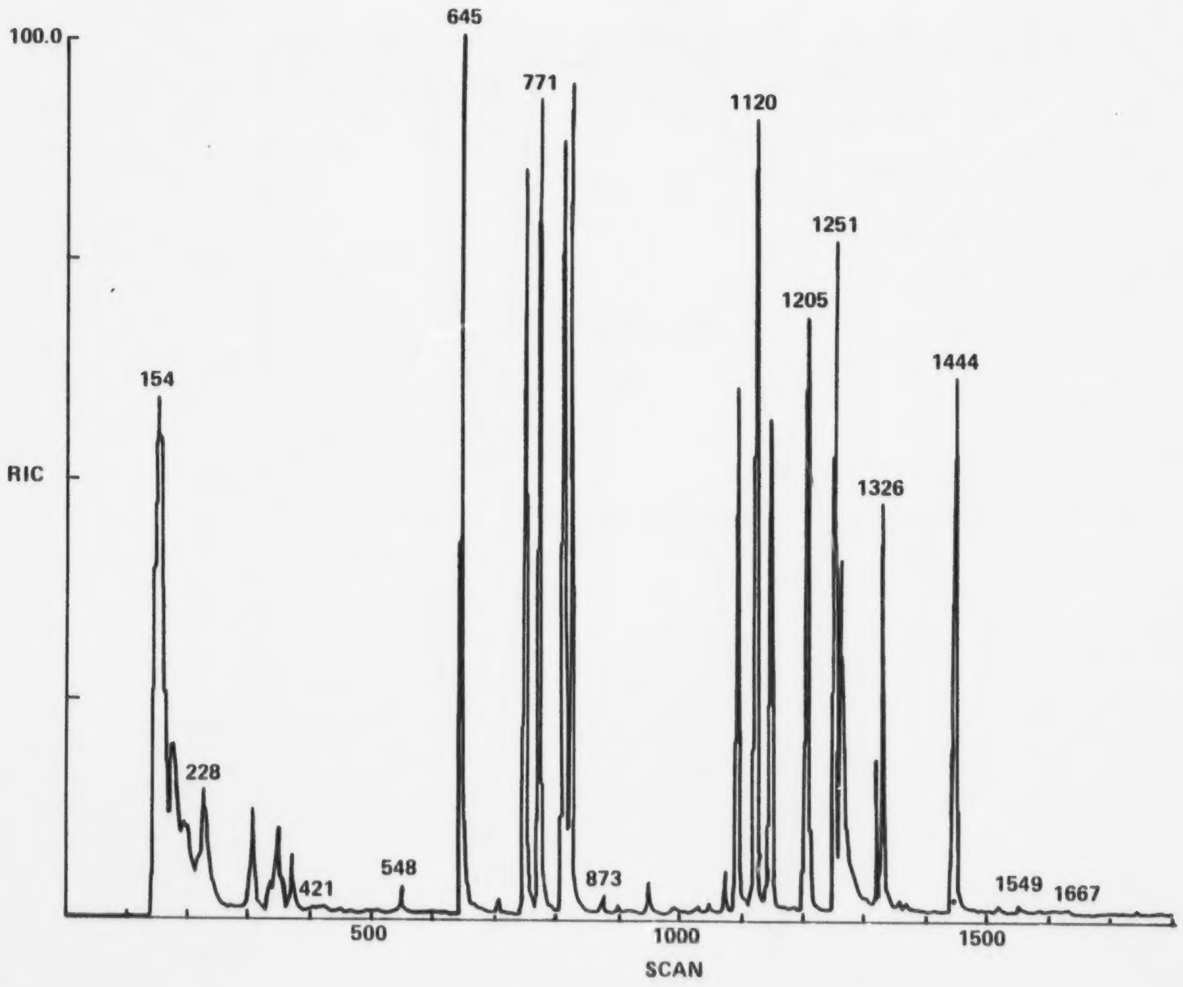


Figure 21. Typical CPMX and RMR run.

22.7 Response Factors

22.7.1 Once the mean response factors have been calculated for the five initial CPMX + RMR and RMR runs, make up a table consisting of the range of numbers within which the response factor must fall for each day of sample analysis.

22.7.2 Determine this range by multiplying the standard deviation by three and subtracting and adding this value to the mean response factor.

NOTE: For example, if the mean response factor of the 97 ion of 1,1,1-Trichloroethane is 1.00 and the standard deviation is 0.20, then the acceptable range would be 0.40-1.60. This range determines if a response factor for a given ion is "in" or "out" of control.

22.7.3 If only one ion of a compound is "in" control, then classify the entire compound "in" control.

22.7.4 Classify a compound as "out" of control only if all ions of a compound are "out" of control.

NOTE: This criterion applies to the target compounds of both the CPMX + RMR and RMR runs.

22.7.5 If the number of compounds "out" of control exceeds seven, analyze another standard cartridge.

22.7.6 If the number of compounds "out" of control is still not acceptable, check the instrument tune, test for an air leak, and establish whether some other problem is present and should be checked.

22.7.7 Tabulate the CPMX + RMR and RMR response factors for each day.

22.8 Estimation of Limits of Detection and Quantifiable Limits

22.8.1 Use an arbitrary noise level of 500 area counts to calculate limits of detection (LOD). Obtain a quantifiable limit of 2000 area counts by multiplying the LOD by four.

22.8.2 Using the raw area counts for a representative CPMX + RMR or RMR compound, make a plot of ng loaded (X-axis) versus area counts (Y-axis) (see Figure 22).

22.8.4 By using a linear regression on the TI-55 calculator compute LOD for each target compound.

NOTE: The quantifiable limit is defined as four times the LOD.

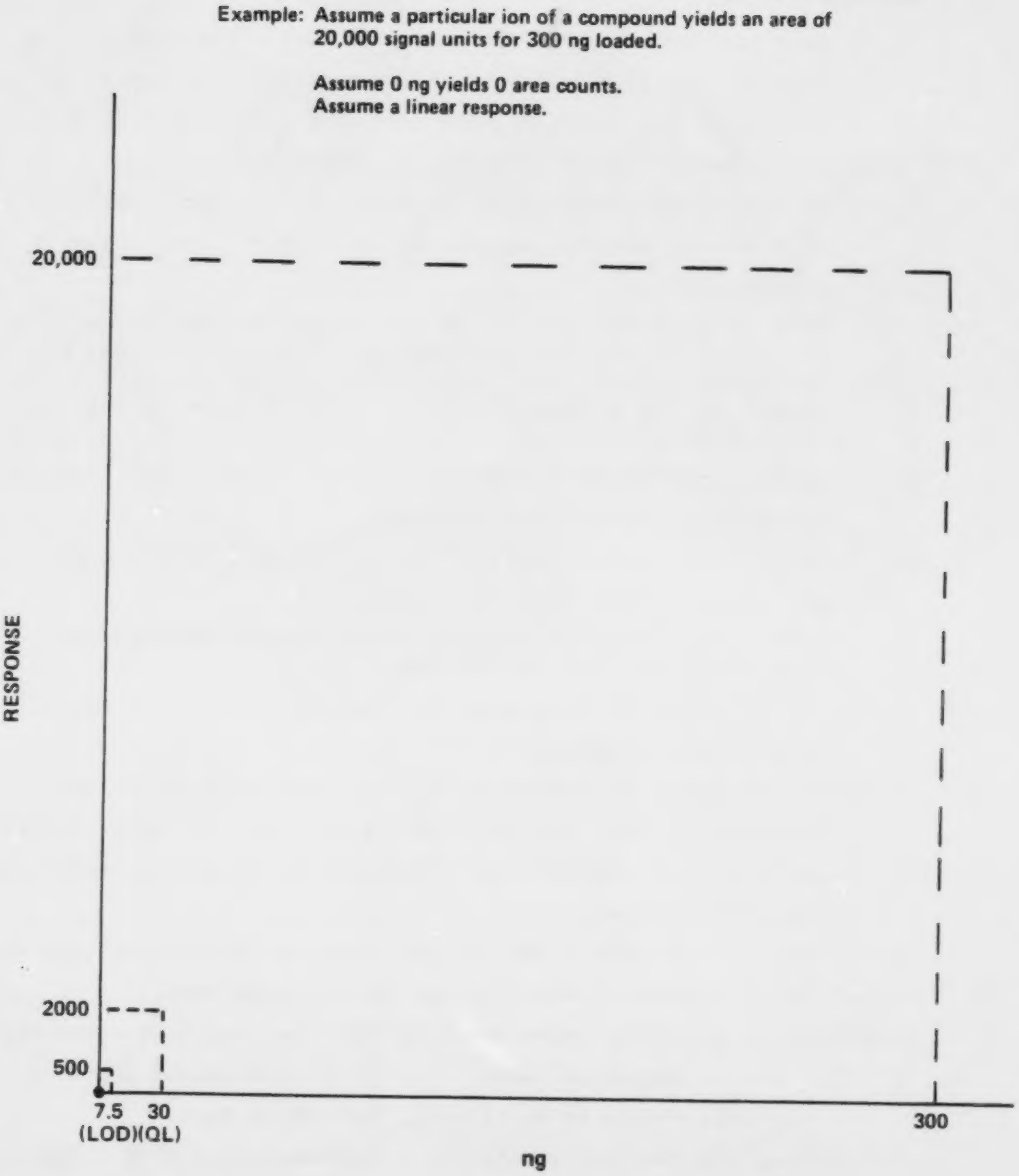


Figure 22. LOD plot.

23.0 DATA BOOKKEEPING

23.1 Log Book

23.1.1 List each sample analyzed on the Finnigan 3300 in the instrument log book, including the date of data acquisition, project number, mode of sample introduction, instrument conditions, type of column used, amount or type of injection, file name, and magnetic tape identification.

23.2 Project Notebook

23.2.1 List each CPMX + RMR, RMR, or sample cartridge run in the project notebook with a file name, date of acquisition, sample name, and GC conditions.

23.2.2 Write information from the Custody Sheet such as batch number of Tenax, temperature, volume, and amount of PFB/PFT loaded.

23.2.3 Leave the Custody sheet for each sample in the notebook until the end of the study.

23.2.4 Tape a chromatogram from each run in the notebook along with the final quantitation report of calculated ng/cart.

23.2.5 In the case of CPMX + RMR and RMR, keep the printout of response factors and tape into project notebook.

23.2.6 For the CPMX + RMR run, keep the column performance calculations of the RIC hardcopies in the project notebook.

24.0 CONCLUSION OF SAMPLE QUANTITATION

24.1 At the conclusion of a study, duplicate all sample quantitation lists and match with the appropriate Custody Sheet.

24.2 Double-check against the instrument log book to assure all samples have been quantitated and copied.

24.3 Relinquish data and Custody Sheets to the appropriate individual.

RTI/ACS-SOP-612-001
December 1983
Revision 0

Standard Operating Procedure for the Preparation of Purge and Trap Calibration Solutions

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Standard Operating Procedure for the Preparation of Purge and Trap Calibration Solutions

RTI/ACS-SOP-612-001

1.0 PURPOSE

Calibration solutions are necessary for the analysis of water samples by purge and trap/gas liquid chromatography. In order to determine the quantity of a given target compound in a water sample, a calibration curve is generated for that compound by analyzing calibration solutions of known concentrations. To ensure proper identification of each target compound, a calibration solution is analyzed daily. A calibration solution is also used for daily measurement of instrumental performance and analytical quality assurance.

2.0 SUMMARY

Two sets of calibration solutions are prepared. The first solutions are designated primary standards. There are two primary standards which are high concentration methanolic solutions of the TEAM target compounds. One solution contains chloroform and bromodichloromethane, and the other solution contains fourteen other TEAM target compounds. Primary standards are prepared by transferring specific volumes of neat compounds to volumetric flasks containing methanol, then diluting to volume. Primary standards are used only to prepare secondary standards.

Secondary standards (working standards) are used for instrument calibration and generation of calibration curves. Secondary standards are prepared at eight different concentrations in methanol by transferring specific volumes of the two primary standards to volumetric flasks containing methanol, then diluting to volume.

The secondary standards are used to spike 25 mL aliquots of contaminant free water over a wide concentration range. All standards are prepared at concentrations so that when 5 μL aliquots of the methanolic secondary standards are added to 25 mL of contaminant-free water, the resulting concentration of target compounds closely resembles the concentrations found in actual drinking water samples.

3.0 APPLICABILITY

The calibration solutions described in this standard operating procedure have been developed specifically for the TEAM project. Concentrations of the standards are based on the analysis procedure used for water samples and have been carefully adjusted after studying historical data obtained from the same instrument. The calibration solutions were developed for the purge and trap determinations of volatiles from 25 mL drinking water samples with the GLC column effluent split five parts to a flame ionization detector and one part to a Hall electrolytic conductivity detector.

4.0 MATERIALS AND REAGENTS

The following list describes all equipment and reagents needed to prepare and store the purge and trap calibration solutions.

4.1 Reagents

- 4.1.1 Methanol, Burdick and Jackson, distilled in glass.
- 4.1.2 TEAM target compounds, see Table 1 for sources and physical data.

4.2 Equipment

- 4.2.1 10 mL glass volumetric flasks with stoppers.
- 4.2.2 100 mL glass volumetric flask with stoppers.
- 4.2.3 Glass beakers.
- 4.2.4 3.7 mL (1.0 dram) glass screw cap vials.
- 4.2.5 7.4 mL (2.0 dram) glass screw cap vials.
- 4.2.6 13 mm Teflon-faced rubber septa, Supelco.
- 4.2.7 15 mm Teflon-faced rubber septa, Supelco.
- 4.2.8 13 mm septa caps, Supelco.

Table 1. TEAM VOLATILE COMPOUNDS ADDED TO PURGE AND TRAP CALIBRATION SOLUTIONS: PHYSICAL DATA AND SOURCES

Compound	Boiling Pt. °C	Molecular Weight	Density (20°C)	Source
vinylidene chloride	31	96.9	1.213	Aldrich (99%)
chloroform	61	119.4	1.492	Aldrich (99%+)
1,1,1-trichloroethane	75	133.4	1.338	Aldrich (97%)
carbon tetrachloride	77	153.8	1.594	Burdick & Jackson (99%+)
benzene	80	78.1	0.874	Burdick & Jackson (99%+)
1,2-dichloroethane	83	99.0	1.256	Aldrich (99%+)
bromodichloromethane	87	163 8	1.980	Aldrich (97%)
trichloroethylene	87	131.4	1.462	Aldrich (99%)
dibromochloromethane	119	208.3	2.451	Alfa Products (98%)
tetrachloroethylene	121	165.8	1.623	Aldrich (99%)
chlorobenzene	132	112.6	1.106	Aldrich (99%)
ethylbenzene	136	106.2	0.867	Aldrich (99%)
p-xylene	138	106.2	0.866	Aldrich (99%)
styrene	145	104.2	0.909	Aldrich (99%)
bromoform	148	252.8	2.890	Eastman Kodak (99%)
m-dichlorobenzene	172	147.0	1.288	Aldrich (98%)

4.2.9 15 mm septa caps, Supelco.

4.2.10 240 mL wide mouth ointment jars with caps.

4.2.11 Labels, 3 x 4 cm.

4.2.12 Transparent tape.

4.2.13 5 µL plunger in needle syringe, SGE, or equivalent.

4.2.14 10 µL Teflon-tipped plunger syringe, Hamilton 1801N or equivalent.

4.2.15 50 µL Teflon-tipped plunger syringe, Precision Sampling or equivalent.

4.2.16 100 µL Teflon-tipped plunger syringe, Precision Sampling or equivalent.

4.2.17 Refrigerator.

4.2.18 Ventilation hood.

4.2.19 Aspirator system to provide vacuum with rubber inlet hose approximately 6 mm i.d.

4.2.20 Thermometer accurate to ±0.5°C.

5.0 PREPARATION OF REAGENTS AND EQUIPMENT

5.1 Checking Reagent Purity and Identity

All of the original TEAM target compounds should be tested for purity and proper identification immediately after acquisition. The two methods generally used are NMR and/or GLC analysis. Purity and identification testing is beyond the scope of this SOP, however, calibration solutions should not be prepared until the structure and purity of all target compounds have been verified.

5.1.1 Methanol Purity Analysis

5.1.1.1 Analyze the methanol used for preparing calibration solutions with the same instrumentation used for water sample analysis (RTI/ACS-SOP-512-001).

5.1.1.2 Set FID attenuation to 8 x 10^{-11}.

5.1.1.3 Set HECD attenuation to 1 and range to 30.

5.1.1.4 Purge 25 mL of Milli-Q water for 15 minutes with the six port valve in the desorb position. Helium flow should be 40 mL/min.

5.1.1.5 Stop helium flow and switch six port valve to purge position by switching valve.

5.1.1.6 Inject 5.0 µL of methanol into the water by inserting the 11.5 cm needle through the septum of the purge flask.

5.1.1.7 Purge the water for 12 minutes at 40 mL/min.

5.1.1.8 After 12 minutes switch the six port valve to desorb, heat the Tenax trap, begin the GC program, and start integrators.

5.1.1.9 Study the integrator data at the end of the run. Identify any peaks eluting at the retention times of target compounds.

5.1.1.10 If any peaks are observed at the retention time of target compounds and have areas greater than 20% of the quantitation limit, obtain new methanol and repeat steps 5.1.1.1 through 5.1.1.9.

5.1.1.11 If no peaks are observed at the retention time of target compounds, the methanol may be used for preparation of calibration solutions.

5.2 Preparation of Containers

All beakers, volumetric flasks, storage vials, and septa must be decontaminated before preparing calibration solutions.

5.2.1 Washing and Labelling

5.2.1.1 Wash glassware in warm soap and water solution. Rinse with Milli-Q water.

5.2.1.2 Bake glassware at 400°C for two hours, then cool.

5.2.1.3 Rinse glassware at least three times with methanol, discarding methanol after each rinse.

5.2.1.4 Dry glassware in a drying oven at 110°C.

5.2.1.5 Wash septa in Milli-Q water and place in ultrasonic bath for 5-10 minutes.

5.2.1.6 Rinse septa twice with methanol, then sonicate for 5-10 minutes. Allow the septa to air dry.

5.2.1.7 Store septa in a clean beaker covered with aluminum foil.

5.2.1.8 Cap vials with septa Teflon face inward.

5.2.1.9 Prepare all labels. Label data must include standard type and designation, solvent, date prepared, and initials of preparer. Example:

TEAM Purge and Trap
Secondary Std.

Ⓑ

In Methanol
6/2/83 KWT

5.2.1.10 The 3.7 mL vials are used for storage of primary standards. Secondary standards are stored in 7.4 mL vials. NOTE: The size difference in vials helps ensure primary and secondary standards are not confused.

5.2.1.11 Prepare three vials for each of the primary standards.

5.2.1.12 Prepare one vial for each secondary standard.

5.2.1.13 Place labels on vials and wrap transparent tape completely around the label and vial.

6.0 PREPARATION OF CALIBRATION SOLUTIONS

Primary calibration standards are prepared by adding specific volumes of target compounds to methanol in volumetric flasks. Target compounds should always be added in the order of least volatile to most volatile to minimize losses. Syringes should be thoroughly rinsed with methanol and vacuum dried between compound deliveries.

Secondary calibration solutions are prepared by adding specific volumes of the two primary standard solutions to methanol in volumetric flasks. The secondary standards are prepared at several concentrations and will be used for generating calibration curves and daily instrumental standards. All secondary standards are prepared so that when 5 µl of standard is added to 25 mL of contaminant free water, the desired water concentration is obtained.

6.1 Preparation of Primary Standards

6.1.1 Calculation of Target Volumes

6.1.1.1 Calculate the volume of each target compound to be added to the two primary standards.

NOTE: Table 2 contains the precalculated volumes for all target compounds and their final concentration in the primary standards.

This section explains how the volumes were obtained.

6.1.1.2 Tetrachloroethylene will be used as an example for volume calculations which may be applied to all other target compounds.

6.1.1.3 Select a nominal concentration to be obtained, 500 ng/µL in methanol in the case of tetrachloroethylene.

6.1.1.4 Select a volume of methanol so that reasonable volumes of target compounds are added. In the case of tetrachloroethylene, the final concentration of 500 ng/µL is easily obtained if a 50 µL syringe is used to deliver the target compound to 100 mL of methanol (50 µL is approximately equal to 50 mg. When 50 mg is added to 100 mL, a concentration of 500 ng/µL is obtained).

6.1.1.5 Calculate the exact volume of tetrachloroethylene needed to produce a 100 mL volume of 500 ng/µL:

(a) 500 ng/µL = 0.5 mg/mL

(b) 0.5 mg/mL x 100 mL = 50 mg target compound needed.

(c) Tetrachloroethylene density = 1.623 ng/µL from Table 1.

(d) $\frac{50 \text{ mg}}{1.623 \text{ mg}/\mu\text{L}} = 30.8\ \mu\text{L}$ tetrachloroethylene.

Table 2. CONCENTRATION OF TEAM COMPOUNDS IN THE WATER PURGE AND TRAP CALIBRATION PRIMARY STANDARDS

Compound	Standard Number	Volumetric Flask Size Used for Dilution	Volume of Neat Compound Added to Volumetric	Concentration (ng/μL)
chloroform	1	10 mL	170 μL	25400
bromodichloromethane	1	10 mL	25.0 μL	4950
vinylidene chloride	2	100 mL	41.0 μL	497
1,1,1-trichloroethane	2	100 mL	37.0 μL	495
carbon tetrachloride	2	100 mL	31.0 μL	494
benzene	2	100 mL	57.0 μL	498
1,2-dichloroethane	2	100 mL	40.0 μL	502
trichloroethylene	2	100 mL	34.0 μL	497
dibromochloromethane	2	100 mL	20.0 μL	490
tetrachloroethylene	2	100 mL	31.0 μL	503
chlorobenzene	2	100 mL	45.0 μL	498
ethylbenzene	2	100 mL	58.0 μL	503
p-xylene	2	100 mL	58.0 μL	502
styrene	2	100 mL	55.0 μL	500
bromoform	2	100 mL	17.0 μL	491
m-dichlorobenzene	2	100 mL	39.0 μL	502

6.1.1.6 Now calculate the concentration obtained when the nearest whole number volume of tetrachloroethylene is added to 100 mL of methanol:

(a) 31.0 µL x 1.623 mg/µL = 50.3 mg tetrachloroethylene.

(b) $\frac{50.3 \text{ mg}}{100 \text{ mL}}$ = 0.503 mg/mL = 503 ng/µL.

6.1.1.7 Calculate the volumes needed for all other target compounds in primary standard number two in a similar fashion.

6.1.1.8 Chloroform and bromodichloromethane must be prepared at much higher concentrations, so a 10 mL volume of methanol is chosen for dilution.

NOTE: In typical drinking water samples the ratio of chloroform to bromodichloromethane to all other compounds is approximately 25:5:1.

6.1.1.9 Calculate the volumes of chloroform and bromodichloromethane for the preparation of primary standard number one as described in 6.1.1.3 through 6.1.1.6.

6.1.2 Equipment Set-Up

6.1.2.1 Place all syringes, volumetric flasks, methanol, and target compounds in a ventilation hood which is 20°C ± 5°C.

6.1.2.2 Allow all equipment temperatures and target compound temperatures to equilibrate for at least 60 minutes.

NOTE: The volumes of target compounds added to the methanol are calculated from density values at 20°C.

6.1.2.3 Set-up aspirator in the hood or nearby.

6.1.2.4 Rinse all syringes with at least 10 full volumes of methanol.

6.1.2.5 Dry syringes by application of vacuum from the aspirator.

6.1.2.6 Label the three 150 mL beakers: "Clean Methanol", "Rinse Methanol", and "Discard Methanol".

6.1.2.7 Add methanol to the "Clean Methanol" beaker.

6.1.2.8 Rinse a 10 mL and 100 mL volumetric flask with methanol. Discard rinse.

6.1.2.9 Fill the volumetric flasks to the base of the neck with methanol and replace stoppers.

6.1.3 Addition of Target Compounds

6.1.3.1 Prepare primary standard number one first.

WARNING

The target compounds are volatile, toxic, and several are suspected carcinogens. Handle the pure compounds only in a ventilation hood. Always wear protective gloves.

6.1.3.2 Add 25.0 μL of bromodichloromethane to the methanol in the 10 mL volumetric flask. Inject under the surface of the methanol.

CAUTION

Always depress the syringe plunger fully and remove the needle immediately afterward. Do not rinse the syringe with methanol in the volumetric flask. Incorrect volume additions may result.

6.1.3.3 Place stopper in volumetric flask.

6.1.3.4 Rinse the syringe with at least 10 full volumes of methanol from the rinse beaker and discard each rinse in the discard beaker.

6.1.3.5 Thoroughly dry the syringe with vacuum from the aspirator.

6.1.3.6 Add 170 μL of chloroform to the 10 mL volumetric flask in two aliquots using the 100 μL syringe. Stopper the volumetric.

6.1.3.7 Clean the syringe as described above.

6.1.3.8 Fill the volumetric flask to the mark by dispensing clean methanol from a Pasteur pipet.

6.1.3.9 Invert the flask five or six times to mix, making sure the stopper is secure.

6.1.3.10 Carefully pour the contents of the 10 mL volumetric flask into the first two 3.4 mL vials, completely filling the vials so that when the vials are capped no headspace remains.

6.1.3.11 Empty the remaining solution into the third 3.4 mL vial and cap tightly.

6.1.3.12 Using the 50 and 100 μL syringes carefully add the fourteen remaining target compounds to the 100 mL volumetric flask. Follow the procedure described in steps 6.1.3.2 through 6.1.3.9. Add compounds in the order of highest to lowest boiling points (Table 1). Clean syringes between each delivery.

6.1.3.13 After all compounds are added and mixed, pour off the methanol contained in the neck of the volumetric flask into the discard beaker.

6.1.3.14 Carefully fill the three 7.4 mL vials and cap tightly. No headspace should remain in the vials.

6.2 Preparation of Secondary Standards

Secondary standards are prepared by adding predetermined volumes of the two primary standards to methanol in volumetric flasks. Each secondary standard is prepared at a different concentration so that water aliquots may be fortified over a wide concentration range using just one fortification volume.

6.2.1 Calculation of Volumes

6.2.1.1 Predetermined volumes of the primary standards which are added to 10 mL volumetric flasks to form secondary standards are listed in Table 3. The rest of this section explains how the volumes are determined.

6.2.1.2 First, determine the range of concentrations for the target compounds in drinking water. For chloroform the range is generally 1 ng/mL to 150 ng/mL. Bromodichloromethane may be found at the limit of detection up to 30 ng/mL. The other target compounds usually are found at concentrations from the limit of detection to 5 ng/mL.

6.2.1.3 Prepare secondary standards so that when 5 µL of the standards are added to 25 mL aliquots of contaminant free water, the resulting concentrations cover the ranges described in 6.2.1.2. Chloroform is used as an example:

(a) 25 mL water x 150 ng/mL chloroform = 3750 ng chloroform.

(b) $\frac{\text{3750 ng chloroform}}{\text{5 µL spike}} = \text{750 ng/µL}$.

6.2.1.4 The same calculations are performed for each compound so that eight standards cover the desired concentration range.

6.2.1.5 After determining the desired concentrations of the secondary standards, calculate the volume of primary standards necessary to achieve those concentrations. Chloroform in the highest concentration secondary standard is used as an example:

(a) Desired secondary standard chloroform concentration = 750 ng/µL.

(b) Volume of secondary standard = 10 mL.

(c) Concentration of chloroform in primary standard = 25400 µg/µL.

(d) 10 mL x 750 µg/mL = 7500 µg chloroform needed.

(e) $\frac{\text{7500 µg}}{\text{25.4 µg/µL}} = \text{295 µL}$ primary standard needed.

6.2.1.6 Calculate the actual concentration in the secondary standard for the most easily delivered volume nearest that determined in 6.2.1.5. For example:

(a) 300 μL of primary standard number one.

(b) 300 μL x 25.4 μg/μL chloroform = 7620 μg.

(c) $\frac{7620\ \mu g\ \text{chloroform}}{10\ mL} = 762\ \mu g/mL = 762\ ng/\mu L.$

6.2.1.7 Refer to Table 3 to determine the volume of primary standard used to prepare the eight secondary standards.

6.2.1.8 Refer to Table 4 to find the secondary standard concentrations (when standards are prepared according to Table 3).

6.2.2 Equipment Set-Up

6.2.2.1 Place all syringes, volumetric flasks, methanol, and primary standards in a ventilation hood which is 20°C ± 5°C.

6.2.2.2 Allow all equipment temperatures to equilibrate for at least 60 minutes.

6.2.2.3 Set up aspirator in the hood or nearby.

6.2.2.4 Rinse all syringes with at least 10 full volumes of methanol.

6.2.2.5 Dry syringes by application of vacuum from the aspirator.

6.2.2.6 Label eight 10 mL volumetric flasks: AA, A, B, C, D, E, F, G.

6.2.2.7 Add methanol to the "Clean Methanol" beaker.

6.2.2.8 Rinse each 10 mL volumetric flask with approximately 5 mL of methanol. Discard rinse.

Table 3. PREPARATION OF PURGE AND TRAP SECONDARY (WORKING) CALIBRATION STANDARDS FROM PRIMARY STANDARDS

Secondary Standard Designation	Volume of Primary Standard No. 1 ($CHCl_3$ + $CHCl_2Br$) to 10 mL Volumetric	Volume of Primary Standard No. 2 (14 compounds) to 10 mL Volumetric
AA	300 µL	750 µL
A	200 µL	500 µL
B	150 µL	200 µL
C	100 µL	100 µL
D	40 µL	50 µL
E	20 µL	25 µL
F	10 µL	10 µL
G	3 µL	5 µL

Table 4. CONCENTRATION OF TEAM COMPOUNDS IN THE WATER PURGE AND TRAP CALIBRATION SECONDARY (WORKING) STANDARDS

Standard Designation:	AA	A	B	C	D	E	F	G
Compound	Concentration (ng/μL)							
vinylidene chloride	37.3	24.3	9.92	4.96	2.48	1.24	0.50	0.25
chloroform	762	508	381	254	102	50.8	25.4	7.60
1,1,1-trichloroethane	37.1	24.8	9.92	4.96	2.48	1.24	0.50	0.25
carbon tetrachloride	37.0	24.7	9.88	4.94	2.47	1.24	0.49	0.25
benzene	37.4	24.9	9.96	4.98	2.49	1.25	0.50	0.25
1,2-dichloroethane	37.6	25.1	10.0	5.02	2.51	1.25	0.50	0.25
bromodichloromethane	148	99.0	74.2	49.5	19.8	9.90	5.00	1.50
trichloroethylene	37.3	24.8	9.92	4.96	2.48	1.24	0.50	0.25
dibromochloromethane	36.8	24.5	9.80	4.90	2.45	1.22	0.49	0.25
tetrachloroethylene	37.7	25.2	10.1	5.04	2.52	1.26	0.50	0.25
chlorobenzene	37.4	24.9	9.96	4.98	2.49	1.25	0.50	0.25
ethylbenzene	37.7	25.2	10.1	5.04	2.52	1.26	0.50	0.25
p-xylene	37.6	25.1	10.0	5.02	2.51	1.25	0.50	0.25
styrene	37.5	25.0	10.0	5.00	2.50	1.25	0.50	0.25
bromoform	36.8	24.6	9.84	4.92	2.46	1.23	0.49	0.25
m-dichlorobenzene	37.6	25.1	10.0	5.02	2.51	1.25	0.50	0.25

6.2.2.9 Fill the volumetric flasks with 8 mL of clean methanol and replace the stoppers.

6.2.3 Addition of Primary Standards

6.2.3.1 Prepare secondary standards in the order of most to least concentrated (AA first and G last).

6.2.3.2 To each volumetric flask add the volumes of primary standards one and two specified in Table 3. Use syringes which most closely match the volume to be delivered.

NOTE: To deliver volumes greater than 100 µL, use the 100 µL syringe for as many transfers as are needed.

6.2.3.4 Fill the volumetric flasks to the 10 mL mark by dispensing clean methanol with a Pasteur pipet.

6.2.3.5 Invert each flask five or six times to mix, making sure the stopper is secure.

6.2.3.6 Carefully pour each secondary standard into the correct, prelabelled vial leaving no headspace. Cap tightly with septa in place.

WARNING

Dispose of all waste solutions remaining in all volumetric flasks in an approved manner.

6.3 Fortification of Water

The secondary standards are used to fortify clean water for generation of calibration curves and running daily instrument performance checks. In all cases the secondary standards have been designed so that when 5 µL of the secondary standards are added to 25 mL of clean water, the desired concentrations are obtained. The water concentrations are designed to cover the range normally found in drinking water samples (Table 5).

6.3.1 Spiking Procedure

6.3.1.1 Fill the water analysis sytem purge flask with 25 mL Milli-Q water and purge according to instructions in SOP "Analysis of Drinking Water by Purge and Trap".

6.3.1.2 Remove the desired concentration secondary standard from the refrigerator and allow it to equilibrate to room temperature for 45 minutes.

Table 5. CONCENTRATION OF TEAM TARGET COMPOUNDS IN WATER FORTIFIED WITH THE SECONDARY (WORKING) CALIBRATION STANDARDS

Secondary Standard:	AA	A	B	C	D	E	F	G
Compound	Concentration When 5 μL of Secondary Standard Is Added to 25 mL water (ng/mL)							
vinylidene chloride	7.46	4.96	1.98	0.99	0.50	0.25	0.10	0.05
chloroform	152	102	76.2	50.8	20.4	10.2	5.10	1.50
1,1,1-trichloroethane	7.42	4.96	1.98	0.99	0.50	0.25	0.10	0.05
carbon tetrachloride	7.40	4.94	1.98	0.99	0.49	0.25	0.10	0.05
benzene	7.48	4.98	1.99	1.00	0.50	0.25	0.10	0.05
1,2-dichloroethane	7.52	5.02	2.01	1.00	0.50	0.25	0.10	0.05
bromodichloromethane	29.6	19.8	14.8	9.90	4.00	2.00	1.00	0.30
trichloroethylene	7.46	4.96	1.98	0.99	0.50	0.25	0.10	0.05
dibromochloromethane	7.36	4.90	1.96	0.98	0.49	0.24	0.10	0.05
tetrachloroethylene	7.54	5.04	2.02	1.01	0.50	0.25	0.10	0.05
chlorobenzene	7.48	4.98	1.99	1.00	0.50	0.25	0.10	0.05
ethylbenzene	7.54	5.04	2.02	1.01	0.50	0.25	0.10	0.05
p-xylene	7.52	5.02	2.01	1.00	0.50	0.25	0.10	0.05
styrene	7.50	5.00	2.00	1.00	0.50	0.25	0.10	0.05
bromoform	7.36	4.92	1.97	0.98	0.49	0.25	0.10	0.05
m-dichlorobenzene	7.52	5.02	2.01	1.00	0.50	0.25	0.10	0.05

6.3.1.3 Clean the 5 μL plunger in needle syringe (11.5 cm needle) with at least 10 full volumes of clean methanol.

6.3.1.4 Stop purging the water and place the six port valve handle to the "purge" position.

6.3.1.5 Remove 5 μL of the secondary standard by inserting the needle through the septum. Remove air bubbles from the syringe by quickly raising and lowering the plunger five or six times. Then with one slow, smooth motion, raise the plunger all the way.

6.3.1.6 Carefully remove the needle from the septum, making sure the needle does not flex.

6.3.1.7 Insert the needle through the septum on the purge flask.

6.3.1.8 Push the needle in until the tip is below the surface of the prepurged water.

6.3.1.9 Depress the syringe plunger all the way and remove needle from the purge flask.

6.3.1.10 Begin purging the water standard.

6.3.1.11 Return the secondary standard to the refrigerator.

6.3.1.12 When the standard has cooled to 4°C replace the septum and return to the refrigerator.

7.0 STORAGE AND LIFETIMES

7.1 Primary Standards

Two full vials of primary standards one and two should be saved after their preparation. Cap the vials tightly with Teflon lined rubber septa. The septa should not be pierced for any reason until the standards are used. Primary standards may be stored up to three months after preparation at 4°C or in a freezer. The vials should have no headspace at 20°C. Store the vials upside down so that any headspace which forms after cooling will be surrounded by glass. Each solution should be discarded after it is used to prepare fresh secondary standards.

7.2 Secondary Standards

Immediately after their preparation, secondary standards should be stored in an ointment jar which is kept in a refrigerator at 4°C. All vials should be tightly capped with Teflon lined rubber septa. There should be no headspace in the vials at 20°C when first prepared. Septa should be replaced after piercing, but the standard must be cooled to 4°C before the septa may be changed.

Secondary standards may be stored up to six weeks at 4°C but may have to be replaced earlier if they show signs of concentration changes. Changes may be determined by comparing daily standard and calibration standard concentrations determined during the course of operations. If any concentration changes are observed outside of instrument variation, the secondary standards must be discarded and new standards prepared. Allowable variation will be determined by comparison with historically accepted variations.

8.0 DOCUMENTATION

Each time new primary and/or secondary standards are prepared, all pertinent information must be entered into the current purge and trap log notebook. The information should include types of standards prepared, concentrations of all target compounds in the standards, date of preparation, name of preparing chemist, sample calculations, and any variation or change incorporated into the procedure.

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Standard Operating Procedure for Preparing Relative Molar Response Tenax Cartridges Using a Permeation System

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Standard Operating Procedure for Preparing Relative Molar Response Tenax Cartridges Using a Permeation System

RTI/ACS-SOP-630-001

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure describes the methodology for loading Tenax cartridges via a dynamic dilution system, hereafter referred to as the "Permeation System" (see Figure 1). A permeation system consists of three main parts: (1) a temperature-controlled chamber containing permeation tubes; (2) a mixing chamber; and (3) two cartridge holders enclosed in an insulated jacket. A stream of nitrogen circulates through the system. The amounts of compounds transported downstream remain constant once the walls of the system become saturated. The amounts of compounds can be determined by measuring the time and the gas flow through the cartridge. One of the most important applications of this system is loading known quantities of compounds onto Tenax cartridges. These cartridges are used to determine the Relative Molar Response (RMR) of the GC/MS/COMP system. The samples prepared for this purpose are designated as RMR cartridges.

This system may be applied to any volatile compound that may be prepared as a permeation tube with a constant permeation rate under controlled conditions. The permeation system may be used to load known quantities of compounds not only onto Tenax, but onto any other sorbent (i.e., charcoal). In addition, with minor modifications this system may be used to load external standards onto a field sample via syringe.

2.0 SUMMARY OF THE METHOD

The following routine shall be followed when Tenax cartridges are loaded with RMR compounds via a permeation system: (1) select the appropriate permeation tubes; (2) determine the loading conditions to be used (i.e., flow rates and duration of loading); (3) equilibrate the system; (4) check the background of the Tenax cartridges; (5) load the

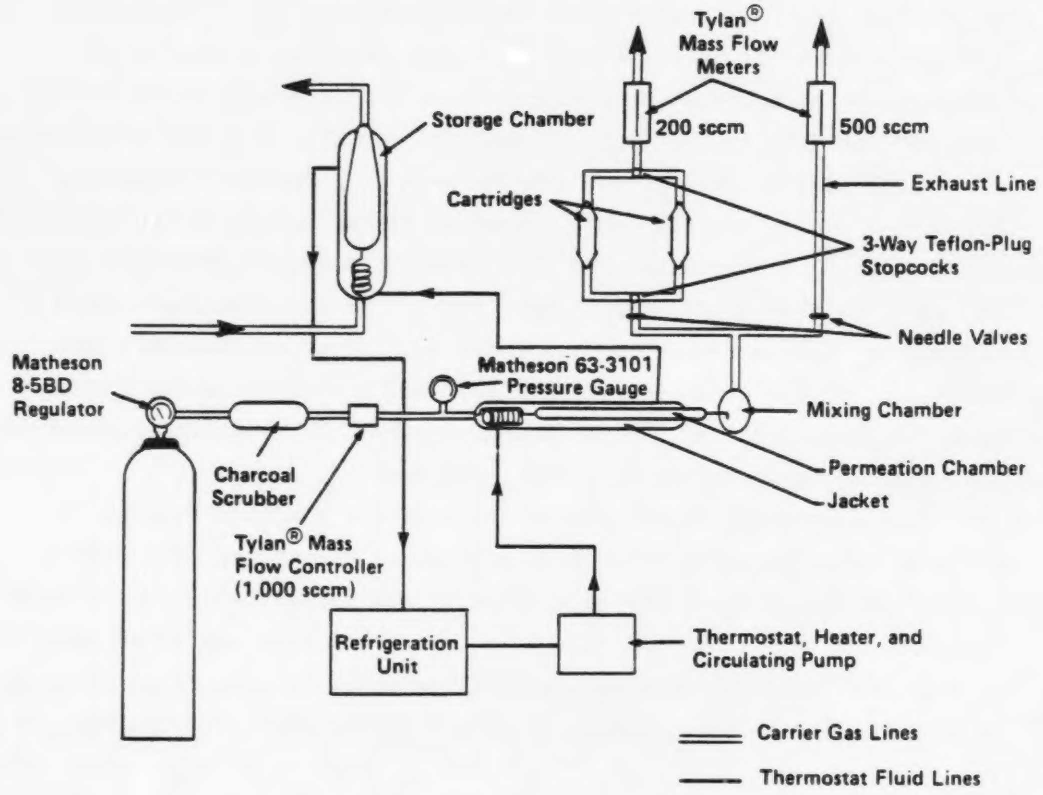
Tylan® Mass Flow Meters
200 sccm
500 sccm
Storage Chamber
Exhaust Line
Cartridges
3-Way Teflon-Plug Stopcocks
Needle Valves
Matheson 8-5BD Regulator
Matheson 63-3101 Pressure Gauge
Mixing Chamber
Permeation Chamber
Jacket
Charcoal Scrubber
Tylan® Mass Flow Controller (1,000 sccm)
Refrigeration Unit
Thermostat, Heater, and Circulating Pump
Carrier Gas Lines
Thermostat Fluid Lines

Figure 1. Permeation system for generating N_2/vapor mixtures.

cartridges; (6) calculate the amounts of compounds loaded; (7) assure the integrity of the loading procedure; and (7) add the external standards. See Appendix I of this SOP for determining permeation rates for the permeation tubes.

3.0 PROCEDURE

3.1 Selection of Permeation Tubes

3.1.1 In a bound RTI notebook, assigned for a specific project, list all the compounds to be loaded.

3.1.2 Check the permeation notebooks (located in the laboratory) to see what permeation tubes are available for each of the target compounds.

NOTE: There is more than one permeation tube available for almost every compound. Each one has been assigned a unique number. The tubes may differ in their permeation rate and/or stability.

3.1.3 For each target compound select the most stable permeation tube whose permeation rate is nearest to ~300 ng/min.

3.1.4 Complete the table below in the RTI notebook, entering the number of the tube to be used and the corresponding permeation rate.

Tube #	Compound	Permeation Rate Mean ± S.D. (CV)

NOTE: The mean permeation rate is calculated using the last five individual permeation rates.

3.2 Determination of the Permeation System Conditions

3.2.1 General Formula

For any compound, calculate the amount loaded onto a cartridge using the following formula:

$$G = (P)\ (t)\left(\frac{F_1}{F_1 + F_2}\right)$$

where:
G = amount loaded (ng)
P = permeation rate (ng/min)
t = time (min)
F_1 = flow rate through the cartridge (mL/min)
F_2 = exhaust flow rate (mL/min)

NOTE: The four variables, G, t, F_1 and F_2, determine the loading conditions. Any three of them may be fixed and the fourth one calculated from the equation.

CAUTION

Do not use permeation tubes with permeation rates below 100 ng/min. Do not use permeation tubes with permeation rates higher than 1×10^5 ng/min. Do not load a cartridge using a cartridge flow below 50 mL/min or above 150 mL/min. Do not operate the system with a total flow below 250 mL/min or above 600 mL/min. Do not sample for less than two minutes. These restrictions decrease the possibility of error.

3.2.2 Calculations

See Examples 1, 2 and 3 for loading specific amounts of compounds within a given range.

Example 1. Objective: To load benzene, chlorobenzene and chloroform onto a cartridge in the range of 200-500 ng per analyte.

Compound	Permeation Rate (ng/min)
benzene	360
chlorobenzene	270
chloroform	520

At a fixed time and gas flow rate through the cartridge, compute the exhaust gas flow rate using the compound with the lowest permeation rate (chlorobenzene).

G = 200 ng Fixing F_1 at 80 mL/min

P = 270 ng/min t to 4 min,

Calculate:

$$G = (P)\ (t) \left(\frac{F_1}{F_1 + F_2} \right)$$

$$F_2 = \frac{(P)\ (t)\ (F_1)}{G} - F_1$$

$$F = \frac{(270\ \text{ng/min})\ (4\ \text{min})\ (80\ \text{mL/min})}{200\ \text{ng}} - 80\ \text{mL/min}$$

$$F_2 = 352\ \text{mL/min}$$

Using these conditions calculate the amount of compound loaded for the tube with the highest permeation rate (chloroform).

$G = ?$
$P = 520$ ng/min
$F_1 = 80$ mL/min
$F_2 = 352$ mL/min
$t = 4$ min

$$G = (P)\ (t) \left(\frac{F_1}{F_1 + F_2} \right)$$

$$G = (520 \text{ ng/min})\ (4 \text{ min})\ \frac{80 \text{ mL/min}}{80 \text{ mL/min} + 352 \text{ mL/min}}$$

$$G = 385 \text{ ng}$$

NOTE: All the values obtained are within the acceptable range.

Example 2. Objective: To load benzene, chlorobenzene and chloroform onto a cartridge in the 100-200 ng per analyte range.

Compound	Permeation Rate (ng/min)
benzene	360
chlorobenzene	270
chloroform	520

Follow the same procedure as in Example 1: (1) consider 100 ng of chlorobenzene (lowest permeation rate); (2) fix the time and flow through the cartridge; and (3) calculate the exhaust flow rate.

$G = 100$ ng
$P = 270$ ng/min
$F_1 = 80$ mL/min
$t = 4$ min
$F_2 = ?$

$$G = (P)\ (t) \left(\frac{F_1}{F_1 + F_2} \right)$$

$$F_2 = \frac{(P)\ (t)\ (F_1)}{G} - F_1$$

$$F_2 = \frac{(270 \text{ ng/min})\ (4 \text{ min})\ (80 \text{ mL/min})}{100 \text{ ng}} - 80 \text{ mL/min}$$

$$F_2 = 784 \text{ mL/min}$$

This exhaust flow rate is higher than the maximum acceptable value (see Section 3.2.1).

t = 2.5 min
F = 60 mL/min
F_2 = ?

$$F_2 = \frac{(P)\ (t)\ (F_1)}{G} - F_1$$

$$F_2 = \frac{(270\ \text{ng/min})\ (2.5\ \text{min}\ (60\ \text{mL/min})}{100\ \text{ng}} = 60\ \text{mL/min}$$

$$F_2 = 345\ \text{mL/min}$$

NOTE: These conditions are acceptable. Now, calculate the amount of compound loaded for the tube with the highest permeation rate (chloroform).

G = ?
P = 520 ng/min
F_1 = 60 mL/min
F_2 = 345 mL/min
g = 2.5 min

$$G = (P)\ (t)\left(\frac{F_1}{F_1 + F_2}\right)$$

$$G = (520\ \text{ng/min})\ (2.5\ \text{min})\ \frac{60\ \text{mL/min}}{60\ \text{mL/min} + 345\ \text{mL/min}}$$

$$G = 193\ \text{ng}$$

NOTE: All the values obtained are within the acceptable range.

Example 3. Objective: To load benzene, chlorobenzene and chloroform onto a cartridge in the range of 500-1000 ng per analyte.

Follow the same procedure as in Examples 1 and 2.

Consider 500 ng for chlorobenzene.

G = 500 ng
P = 270 ng/min
F_1 = 80 mL/min
F_2 = ?
t = 4 min

$$G = (P)\ (t)\left(\frac{F_1}{F_1 + F_2}\right)$$

$$F_2 = \frac{(P)\ (t)\ (F_1)}{G} - F_1$$

$$F_2 = \frac{(270\ \text{ng/min})\ (4\ \text{min})\ (80\ \text{mL/min})}{500\ \text{ng}} - 80\ \text{mL/min}$$

$$F_2 = 93\ \text{mL/min}$$

The total flow rate (173 mL/min) is below the acceptable level (see 3.2.1 CAUTION).

Now consider the same flow rate through the cartridge but for twice the length of time.

G = 500 ng
P = 270 (ng/min)
F_1 = 80 mL/min
t = 8 min
F_2 = ?

$$F_2 = \frac{(P)\ (t)\ (F_1)}{G} - F_1$$

$$F_2 = \frac{(270\ \text{ng/min})\ (8\ \text{min})\ (80\ \text{mL/min})}{500\ \text{ng}} - 80\ \text{mL/min}$$

$$F_2 = 266\ \text{mL/min}$$

NOTE: This exhaust rate is acceptable.

Finally, calculate the amount of compound loaded for the tube with the highest permeation rate.

G = ?
P = 520 (ng/min)
F_1 = 80 mL/min
t = 8 min
F_2 = 266 mL/min

$$G = (P)\ (t) \left(\frac{F_1}{F_1 + F_2} \right)$$

$$G = (520\ \text{ng/min})\ (8\ \text{min}) \left(\frac{80\ \text{mL/min}}{80\ \text{mL/min} + 266\ \text{mL/min}} \right)$$

$$G = 962\ \text{ng}$$

NOTE: All the values obtained are within the acceptable range.

3.3 Materials and Equipment

3.3.1 Permeation system (see Figure 1)
3.3.2 Nitrogen gas (99.995% purity)
3.3.3 Nylon gloves
3.3.4 Long glass hook (for retrieving permeation tubes)
3.3.5 Permeation tubes
3.3.6 Kimwipes®
3.3.7 Stopwatch

3.4 Equilibration of the Permeation System

3.4.1 Turn off the nitrogen flow in the storage chambers (see Figure 2).

3.4.2 Locate the chambers in which the selected permeation tubes are stored.

WARNING

Open one storage chamber at a time. They contain toxic materials that are dangerous to your health.

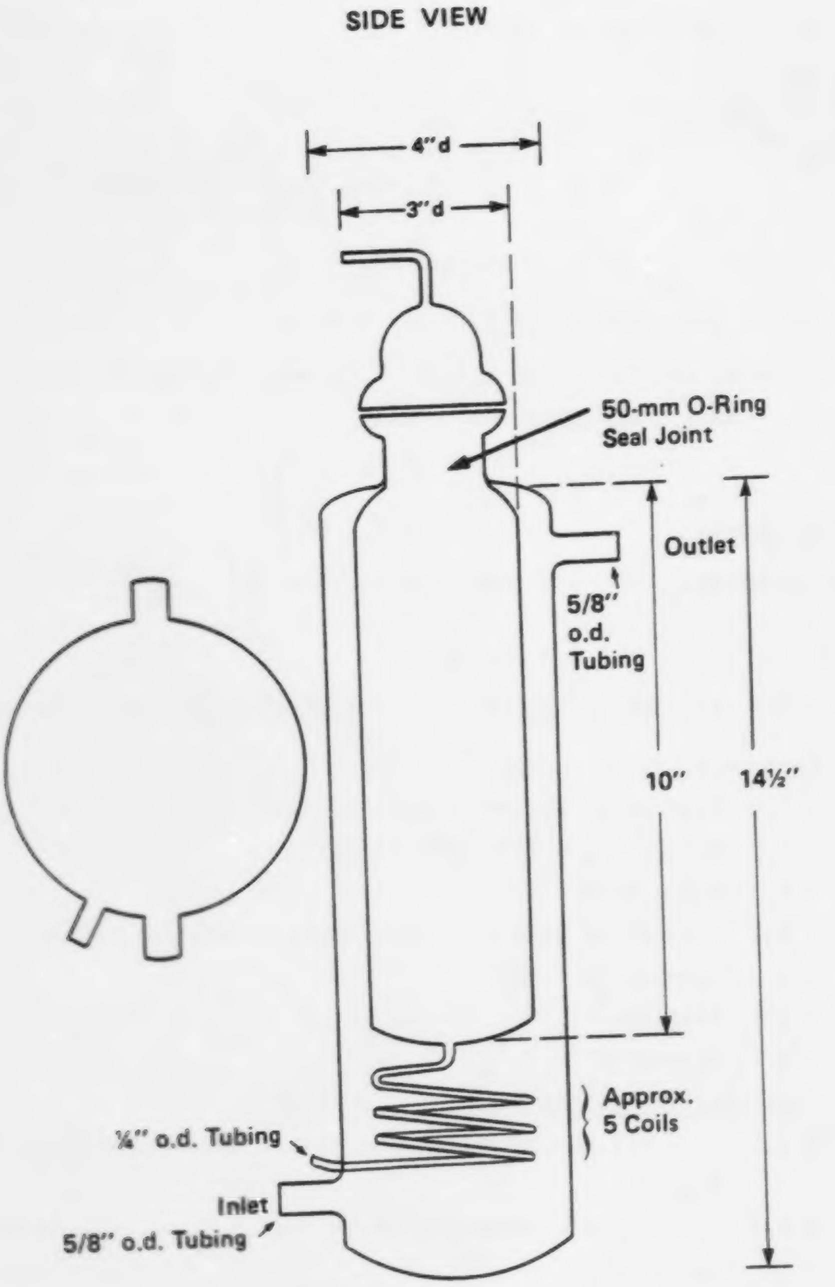
SIDE VIEW
4"d
3"d
50-mm O-Ring
Seal Joint
Outlet
5/8"
o.d.
Tubing
10"
14½"
Approx.
5 Coils
¼" o.d. Tubing
Inlet
5/8" o.d. Tubing

Figure 2. Storage chamber.

3.4.3 Open the loading chamber as follows:

3.4.3.1 Lower the jack which supports the chamber.

3.4.3.2 Remove the clamp securing the two chamber sections.

3.4.3.3 Carefully open the loading chamber by moving the downstream section to the rear.

3.4.3.4 Use jack to support the section.

3.4.3.5 Remove the "O" ring.

WARNING

Failure to wear nylon gloves when handling permeation tubes may result in skin damage and/or contamination of the pereation tubes.

3.4.4 With a long glass hook, remove the selected permeation tubes from the storage chamber and transfer immediately to the loading chamber.

WARNING

Perform this operation as quickly as possible. The tubes contain toxic materials, some of which are cancer-suspect agents.

3.4.5 After the last permeation tube has been transferred, reposition the "O" ring and reconnect the two sections of the loading chamber.

3.4.6 Attach the clamp.

3.4.7 Close and secure the storage chambers. Initiate nitrogen flow by opening the needle valve connected to the "house" nitrogen line.

CAUTION

Ensure that the rotometer indicates sufficient flow rate to distribute approximately 500 mL/min through each storage chamber.

3.4.8 Turn on the read-out box.

NOTE: The read-out box displays the measurements of the mass flow meter and the mass flow controllers. The labels on the box indicate the proper channels.

3.4.9 Ensure that the pressure regulator on the nitrogen tank is fully open (counter-clockwise) (i.e., no pressure).

3.4.10 Turn on the nitrogen gas.

CAUTION

Ensure that the read-out box power is on before any nitrogen circulates through the system, otherwise, the controller will be fully open and the excessive nitrogen flow may damage the system.

3.4.11 Carefully, increase the nitrogen pressure to the permeation system by rotating the pressure regulator T-handle clockwise, observing the gauge in the system at all times. Do not allow the pressure to exceed 10 inches of water.

3.4.12 Set the outlet pressure at the nitrogen cylinder to 20 psi.

3.4.13 Rotate the two stopcocks to direct the nitrogen flow to the side where the Tenax cartridge will be loaded (see Figure 1).

NOTE: When a cartridge is not being loaded, a dummy cartridge is placed in the loading position.

3.4.14 Turn the knob on the read-out box to the total flow rate channel. Adjust the total flow rate with the appropriate controller adjustment knob.

3.4.15 Stabilize the flow rates for approximately two minutes.

3.4.16 Turn the knob to the cartridge flow rate channel. Adjust the nitrogen flow rate through the cartridge with the proper needle valve.

3.4.17 Turn the knob to the exhaust flow rate channel.

3.4.18 Read the exhaust flow rate.

NOTE: The exhaust flow rate is equal to the total flow rate minus the flow rate through the cartridge.

CAUTION

If the total flow rate does not equal the sum of the exhaust flow rate and the cartridge flow rate, a gas leak should be suspected. Ensure that the "O" ring is properly seated and the clamp is secure.

3.4.19 Allow the system to equilibrate for 90 minutes before loading any cartridges.

3.4.20 Recheck loading conditions. Read again the flow rates on the different channels.

3.5 Loading of the Tenax Cartridges

Be sure the background of the Tenax cartridges is acceptable before loading them. Follow Background Check section of SOP-320-001.

3.5.1 Direct the nitrogen flow to the side that will not be used for loading cartridges (see Figure 1) by turning the two stopcocks clockwise, simultaneously.

3.5.2 Loosen the Cajon® fittings and insert the Tenax cartridge with the bottom part down (see Figure 3).

WARNING

Use Kimwipes® when handling the cartridge to avoid contamination.

3.5.3 Tighten the bottom fitting first, then the upper one.

3.5.4 Rotate the two stopcocks clockwise, simultaneously, to direct the gas flow through the cartridge.

3.5.5 Concurrently, start the stopwatch.

3.5.6 Turn the knob on the read-out box to the cartridge flow rate channel. Record this flow rate in the notebook.

3.5.7 Turn the knob to the channel displaying the exhaust flow rate. Record this flow rate in the notebook.

3.5.8 Calculate the time needed to load the amounts desired as follows:

$$t = \left(\frac{G}{P}\right)\left(\frac{F_1 \quad F_2}{F_1}\right)$$

(see Section 3.2.1 for definitions).

Perform the calculation based only on the compound whose permeation tube has either the highest or lowest permeation rate.

3.5.9 At the calculated time rotate the two stopcocks to direct gas flow away from the cartridge being loaded.

3.5.10 Handling the cartridge with a Kimwipe®, loosen the Cajon® fittings, the bottom side first.

3.5.11 Remove the cartridge and return it to its culture tube. Seal the tube.

3.5.12 Label the tube. Include the following information: (1) project number; (2) type of mixture loaded; (3) a number indicating the order of loading; and (4) the date. For example:

2190-34-RMR-03
5-28-83

3.5.13 Prepare a Chain-of-Custody sheet (COC) sheet with the following information:

CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute, Analytical Sciences Division
Chemistry and Life Sciences Group, Research Triangle Park, NC 27709

SAMPLE CODE 2190-34-RMR-03 5-28-83	**TENAX BATCH:**	**VOLUME COLLECTED:**
SAMPLE TYPE: TENAX	**TEMPERATURE (°F)**	**VOLUME ANALYZED:**

RELIN-QUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	(Your initials)		Date of Preparation	RMR mxt. from permeation system.
(Your initials)				

3.5.14 Analyze by GC/FID the first cartridge loaded, according to the conditions specified in the QA section of SOP XXX-YYY (Preparation of Tenax).

3.5.15 Refer to the Quality Assurance section in this SOP (Section 3.10) for more details.

3.5.16 To load another cartridge follow instructions in Steps 3.5.2 to 3.5.13.

3.5.17 To change the flow rates to load different amounts of compounds with the same permeation tubes follow instructions in Steps 3.4.14 to 3.4.18.

3.5.18 Allow the system to equilibrate for fifteen minutes before loading.

3.5.19 To load a new cartridge, follow instructions in Steps 3.5.2 to 3.5.13.

3.6 Turning Off the Permeation System

3.6.1 Open the pressure regulator slowly (counter-clockwise) until fully open.

3.6.2 When the back pressure gauge indicates no pressure, open the loading chamber as follows: (1) lower the jack; (2) remove the clamp; (3) carefully move the upper part of the system back; (4) use jack to support the system; and (5) remove the "O" ring.

3.6.3 Turn off the nitrogen flow in the storage chambers.

WARNING

Failure to wear nylon gloves when handling permeation tubes may cause skin damage and/or contamination of the permeation tubes.

3.6.4 With a long glass hook remove one tube at a time, from the loading chamber and transfer immediately to the respective storage chamber.

3.6.5 After the last tube has been transferred reclamp the storage chambers.

3.6.6 With the needle valve that is connected to the "house" nitrogen line, turn the nitrogen gas on.

3.6.7 Reposition the "O" ring on the loading chamber and move the upper part of the system back to the normal position.

3.6.8 Attach the clamp.

3.6.9 Turn off the read-out box.

3.7 Storage of the RMR Cartridges

Store the RMR cartridges in a sealed paint can in the freezer until they are ready to be loaded with the external standards (see SOP-XXX-YYY).

3.8 Calculations of the Amounts Loaded

3.8.1 For each compound, calculate the amount loaded onto a cartridge using the general formula:

$$G = \left(P\right)\left(t\right)\left(\frac{F_1}{F_1 + F_2}\right)$$

(see Section 3.2.1 for definitions).

3.8.2 Prepare a table to send to GC/MS/COMP that includes: (1) the number of RMR cartridges prepared; (2) the date of preparation; (3) the compounds names; (4) the amounts (in ng/cartridge) loaded for each compound; and (5) the project number (see Table 1 as an example).

3.8.3 Affix a copy of Table 1 in the RTI notebook.

3.9 Loading of External Standards onto RMR Cartridges

For the loading of external standards onto RMR cartridges refer to SOP-630-003.

3.10 Quality Assurance of the RMR Cartridges

3.10.1 Analyze the first cartridge loaded using a Varian 3700 gas chromatograph equipped with a thermal desorption injection unit and a flame ionization detector and interfaced to a Varian CDS 111 integrator.

3.10.2 Analyze the cartridge using the following instrument conditions:

3.10.2.1 Column: Fused silica capillary column (60 m x 0.25 μm), Liq. phase DB1; or an equivalent column

3.10.2.2 Detector temp: 300°C

3.10.2.3 Injector temp: 280°C

3.10.2.4 Desorption unit temp: 260°C

3.10.2.5 Temp. Program: 30°C (2 min), 4°C/min to 220° (10 min)

3.10.2.6 Attenuation: 128×10^{-12}

3.10.2.7 Recorder chart speed: 1 cm/min

3.10.2.8 He carrier flow: 1.2 mL/min

3.10.2.9 H_2 flow to FID: 30 mL/min

3.10.2.10 Air flow to FID: 300 mL/min

3.10.2.11 Make-up gas: ~30 mL/min

3.10.3 For each compound, calculate the instrumental response factor using the following formula:

$$\text{Response Factor} = \frac{\text{Area Counts}}{\text{ng loaded}}$$

3.10.4 Compare the calculated response factor with historical response factor generated under identical conditions.

NOTE: The historical data is stored as the mean response factor and standard deviation for each compound. A response factor is considered out-of-control when it deviates from the mean response factor by more than three standard deviations (3σ). This data is stored in a bound RTI notebook, assigned only for the instrument.

3.10.5 If less than two compounds are out-of-control, load the RMR cartridges for GC/MS/COMP.

NOTE: Chloroform is not included in this analysis due to its low response on FID.

3.10.6 If two or more compounds are out-of-control, load a second cartridge and analyze it.

3.10.7 If the second one is in control proceed with the loading.

3.10.8 If the second RMR cartridge is out-of-control, load and analyze a Column Performance Evaluation (CPM) cartridge. See SOP-630-002 for instructions.

NOTE: The analysis of a CPM cartridge provides a check on the instrument conditions.

3.10.9 If the CPM cartridge is in control, check the permeation system for leaks.

CAUTION

Positive results from a CPM analysis indicates a failure in the permeation system. Be sure the problem is solved before loading the RMR cartridges for GC/MS/COMP.

3.10.10 If the CPM cartridge is out-of-control, troubleshoot the instrument before proceeding with the loading.

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Revision 0

Standard Operating Procedure for Preparing Relative Molar Response and Column Performance Evaluation Tenax Cartridges Using a Flash Evaporation System

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Vice-President, RTI/ACS Date

Standard Operating Procedure for Preparing Relative Molar Response and Column Performance Evaluation Tenax Cartridges Using a Flash Evaporation System

RTI/ACS-SOP-630-002

1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is two-fold: 1) to describe the methodology for preparing column performance evaluation (CPMX) and relative molar response (RMR) solutions, and; 2) to describe the loading of Tenax GC cartridges with volatile organic compounds and the CPMX components using a flash evaporation system.

This method is applicable to organic compounds whose breakthrough volumes are significantly greater than the diluting solvent. Furthermore, the retention of the solvent by the Tenax must be minimal. Breakthrough volume is defined as that volume of gas required to elute 50% of a compound introduced into one end of a cartridge.

2.0 SUMMARY

The main body of the Flash Evaporation System consists of a glass tube with an injection port. The tube is maintained at a temperature of 250°C with heating tape connected to a variable transformer.

A stream of helium gas transports any solution injected into the port to a sorbent cartridge connected on line (see Figure 1).

3.0 PROCEDURE

3.1 Preparation of a CPMX Solution

A CPMX solution contains compounds from different chemical classes. This solution is used to determine mass calibration and intensity and chromatographic performance of the GC/MS/COMP system.

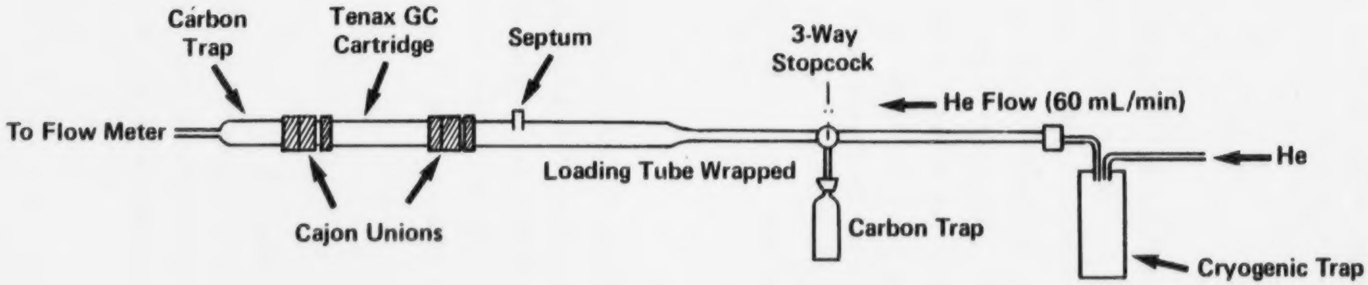
Carbon Trap
Tenax GC Cartridge
Septum
3-Way Stopcock
He Flow (60 mL/min)
To Flow Meter
He
Loading Tube Wrapped
Cajon Unions
Carbon Trap
Cryogenic Trap

Figure 1. Flash evaporation system.

3.1.1 Materials and Instrumentation

3.1.1.1 Capillary pipet, 50 μL or 50 μL syringe

3.1.1.2 Capillary pipet, 100 μL or 100 μL syringe

3.1.1.3 Volumetric flask, 5 mL

3.1.1.4 Volumetric flask, 10 mL

3.1.1.5 Disposable gloves

3.1.1.6 Small spatula

3.1.1.7 Microbalance

3.1.1.8 Pasteur pipets

3.1.1.9 Small aluminum weigh boat

3.1.1.10 Vials with screw cap (open top), 4 mL

3.1.1.11 Silicone rubber septum, Teflon-faced

3.1.2 Reagents

All reagents used are analytical reagent grade (purity >97%).

3.1.2.1 Methyl alcohol, B&J distilled in glass

3.1.2.2 *n*-Octane

3.1.2.3 *n*-Decane

3.1.2.4 5-Nonanone

3.1.2.5 Acetophenone

3.1.2.6 2,6-Dimethylphenol

3.1.2.7 2,6-Dimethylaniline

3.1.2.8 *p*-Xylene

3.1.2.9 *o*-Xylene

3.1.2.10 Ethylbenzene

3.1.2.11 1-Octanol

3.1.3 Procedure

3.1.3.1 In a bound RTI notebook, make a list of the compounds to be used and their respective densities.

3.1.3.2 Calculate the amount required to prepare a stock solution of ~15-20 μg/μL per analyte in a 5 mL volumetric flask.

3.1.3.3 Prepare a table that includes:

Compound Name	Density (mg/μL)	μL Transferred	mg/5 mL	μg/μL

3.1.3.4 Ensure that all the compounds are on hand before preparation of the solution.

WARNING

Use gloves and a hood when handling these chemicals as they may be dangerous to your health.

3.1.3.5 Using a microbalance, weigh the 2,6-dimethylphenol in an aluminum weigh boat and transfer it to the volumetric flask.

WARNING

Do not spend too much time weighing this compound. It is toxic and hygroscopic.

3.1.3.6 Add ~0.5 mL of methyl alcohol to the volumetric flask.

3.1.3.7 Aliquot all liquid compounds in a hood, one at the time, using capillary pipets or syringes.

3.1.3.8 Rinse the walls of the flask with methyl alcohol using a Pasteur pipet to avoid volatility losses.

3.1.3.9 After the last compound has been added, stopper the volumetric flask and invert it five times to mix. Dilute to volume with methyl alcohol.

3.1.3.10 Label the flask as CPMX stock solution. Include the date of preparation and your initials.

3.1.3.11 Mix well. Transfer 100 µL of the stock solution to a 10 mL volumetric flask.

3.1.3.12 Dilute to volume. Mix well and transfer to three screw-cap vials with silicone septa.

3.1.3.13 Label then as CPMX solutions 1, 2, and 3. Include the date of preparation, your initials and the page of the notebook where all the information was recorded.

3.1.3.14 Transfer the stock solution to a screw-cap vial and record all pertinent information.

3.1.3.15 Store all standard solutions at 4°C protected from the light.

3.1.3.16 Open a new vial of the diluted solution every 6 weeks.

3.1.3.17 Use the concentrated stock solution to prepare other dilutions for up to 6 months.

3.2 Preparation of a CPMX/RMR Solution

A CPMX/RMR solution contains all the compounds indicated in the CPMX solution plus all the target compounds of interest for which no permeation tubes are available.

NOTE: Each morning of operation, the Mass Spectrometer operator runs a CPMX/RMR cartridge before beginning the analysis of air or breath samples. This first run is used to evaluate the conditions of the GC capillary column and to calculate the Relative Molar Response of the Mass Spectrometer to the different compounds.

3.2.1 Materials and Instrumentation

3.2.1.1 Capillary pipet, 50 µL or 50 µL syringe

3.2.1.2 Capillary pipet, 100 µL or 100 µL syringe

3.2.1.3 Volumetric flask, 5 mL

3.2.1.4 Volumetric flask, 10 mL

3.2.1.5 Disposable gloves

3.2.1.6 Small spatula

3.2.1.7 Microbalance

3.2.1.8 Pasteur pipets

3.2.1.9 Small aluminum weigh boat

3.2.1.10 Vials with screw cap (open top), 4 mL

3.2.1.11 Silicone rubber septum, Teflon-faced

3.2.2 Reagents

3.2.2.1 Neat compounds (purity >97%)

3.2.2.2 Methyl alcohol, B&J distilled in glass

3.2.3 Procedure

3.2.3.1 To prepare the stock RMR solution, follow the same general instructions as for the CPMX solution (steps 3.1.3.1 to 3.1.3.9 with the exception of step 3.1.3.5).

3.2.3.2 Label the flask as RMR stock solution. Include the date of preparation and your initials.

3.2.3.3 Transfer 100 µL of the RMR stock solution to a 10 mL volumetric flask.

3.2.3.4 Add 100 µL of the CPMX stock solution.

3.2.3.5 Dilute to volume. Mix well and transfer to three screw-cap vials with silicone septa.

3.2.3.6 Label them as CPMX/RMR solutions. Include the date of preparation, your initials and the page of the notebook where it was recorded.

3.2.3.7 Transfer the stock solution to a screw-cap vial and record all pertinent information.

3.2.3.8 Store the vials at 4°C.

3.2.3.9 Use a new vial every 6 weeks.

3.3 Use of the Flash Evaporation System

3.3.1 Materials and Apparatus

3.3.1.1 Flash evaporation system (see Figure 1)

3.3.1.2 Helium, 2500 psi cylinder (guaranteed 99.995%), with regulator

3.3.1.3 Stopwatch

3.3.1.4 Kimwipes®

3.3.1.5 Hamilton microsyringe, 5 µL

3.3.1.6 Septum, half hole type, low bleed-1

3.3.1.7 Tweezers

3.3.1.8 Insulated gloves

3.3.1.9 Methyl alcohol, B&J distilled in glass

3.3.1.10 Silicone rubber septum, Teflon-faced

3.3.2 Loading of a Tenax GC Cartridge

CAUTION

Be sure the Tenax cartridges are clean before loading them. Follow QA Section of SOP-860-001.

3.3.2.1 Turn on helium gas.

3.3.2.2 After 2 or 3 minutes, fill the cryogenic trap with liquid nitrogen.

WARNING

Introducing the liquid nitrogen before the helium is flowing will cause any water that may be present in the line to freeze and prevent passage of the helium through the system.

3.3.2.3 Rotate the stopcock to direct gas flow through the loading chamber.

3.3.2.4 Adjust the flow with the needle value to approximately 60 mL/min, using the stopwatch and bubble meter.

3.3.2.5 Calculate the flow in mL/min according to the equation:

$$\text{Flow} = \frac{\text{volume (mL)}}{\text{time (sec)}} \times \frac{60\ \text{sec}}{1\ \text{min}}$$

3.3.2.6 Repeat the measurement twice.

3.3.2.7 Rinse the syringe several times with clean methyl alcohol.

3.3.2.8 Put the Tenax cartridge on line with the bottom part toward the loading chamber (see Figure 2).

CAUTION

To avoid contamination, always handle the Tenax cartridge with Kimwipes® or clean nylon gloves.

3.3.2.9 Inject the appropriate volume of the solution to be loaded (normally 2 µL) through the septum, holding the needle in place for 15 seconds.

3.3.2.10 At the same time, start the stopwatch.

3.3.2.11 Pass helium through the cartridge at ~60 mL/min for 12 min (~700 mL total).

NOTE: The breakthrough volume for methyl alcohol is 600 mL.

3.3.2.12 Turn the stopcock to the exhaust position to stop the flow through the system.

3.3.2.13 Return the cartridge to the culture tube.

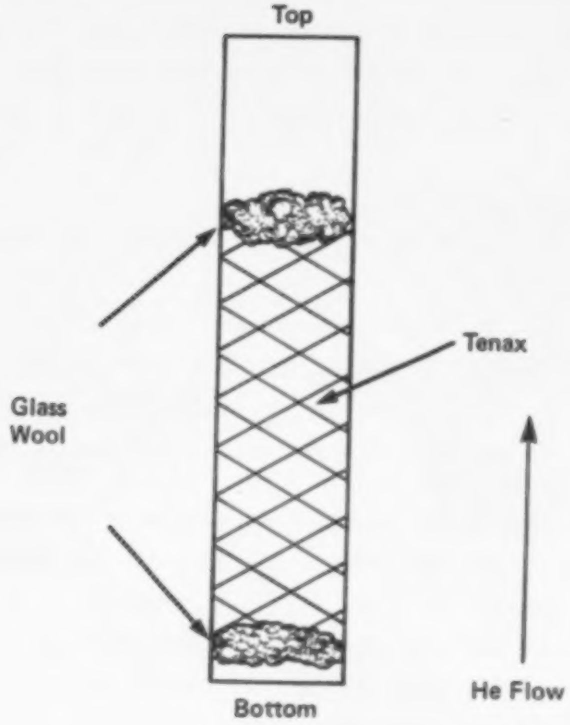
Top
Glass Wool
Tenax
He Flow
Bottom

Figure 2. Tenax cartridge.

3.3.2.14 Label the culture tube. Include the project number, solution loaded, a number indicating the order of loading, and the data; for example:

2190-34-CPMX/RMR-02
5/20/83

3.3.2.15 Prepare a Chain-of-Custody sheet (COC sheet) with the following information as shown in Figure 3.

3.3.2.16 To load another cartridge, rotate the stopcock to direct the flow through the system.

3.3.2.17 Follow instructions in steps 3.3.2.7 to 3.3.2.15.

3.3.2.18 To turn off the system, close the helium tank. Maintain the temperature of the system at 250°C.

3.3.2.19 Change the Teflon-silicone septum in the solution vial.

3.3.2.20 Store the solution at 4°C protected from the light.

3.3.2.21 Store the loaded Tenax cartridges in a sealed paint can in the freezer until they are ready to be loaded with the external standards (see SOP-860-001).

3.4 Maintenance of the Flash Evaporation System

3.4.1 Replace the low-bleed septum at the end of the day.

3.4.2 To replace the septum, support the glass tube (250°C) and remove the septum carefully with a pair of tweezers.

3.4.3 Insert a new septum and let it bake overnight before use.

3.4.4 Replace the activated charcoal in the carbon traps every six months.

3.5 Quality Assurance

After a group of cartridges have been loaded, one or two shall be analyzed by GC/FID according to the conditions specified in the QA section of SOP-320-001 (Preparation of Tenax).

CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute, Analytical Sciences Division
Chemistry and Life Sciences Group, Research Triangle Park, NC 27709

SAMPLE CODE 2190-34-CPMX/RMR-02 5/20/83	TENAX BATCH:	VOLUME COLLECTED:
SAMPLE TYPE: TENAX	TEMPERATURE (°F)	VOLUME ANALYZED:

RELINQUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	your initials		date of preparation	2 µL CPMx/RMR sol. from FES
your initials				

Figure 3. Chain-of-custody sheet.

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December 1983
Revision 0

Standard Operating Procedure for Loading External Standards on Tenax Cartridges Via Injection Using a Permeation System

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Standard Operating Procedure for Loading External Standards on Tenax Cartridges Via Injection Using a Permeation System

RTI/ACS-SOP-630-003

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure describes the methodology for loading the external standards, perfluorobenzene (PFB) and perfluorotoluene (PFT) onto Tenax cartridges. This is performed by syringe injection from a permeation system. A permeation system (see Figure 1) consists of three main parts: 1) a temperature-controlled chamber containing permeation tubes; 2) a mixing chamber; and 3) two holders enclosed in an insulated jacket. One side is a cartridge holder and the other side contains a loading tube.

A stream of nitrogen flows through the system. The amounts of compounds transported downstream remain constant once the walls of the system become equilibrated with the compounds to be loaded. The amounts of external standards injected onto a Tenax cartridge are determined by measuring the total flow through the system and the volume of gas used. This Standard Procedure is used for loading external standards onto field samples, quality control (QC) samples, relative molar response (RMR) cartridges and system performance mixture (CPMX/RMR) cartridges.

Perfluorobenzene (PFB) is used as an external standard to determine the relative retention times and the relative response factors of the compounds to be quantitated. Perfluorotoluene (PFT) is employed for determining instrument stability as related to mass resolution and relative ion abundance under GC conditions.

2.0 SUMMARY OF THE METHOD

The following routine shall be followed when Tenax cartridges are loaded with perfluorobenzene and perfluorotoluene as external standards, by syringe injection from a permeation system: 1) select the permeation tubes; 2) prepare the permeation system; 3) select the cartridges to be

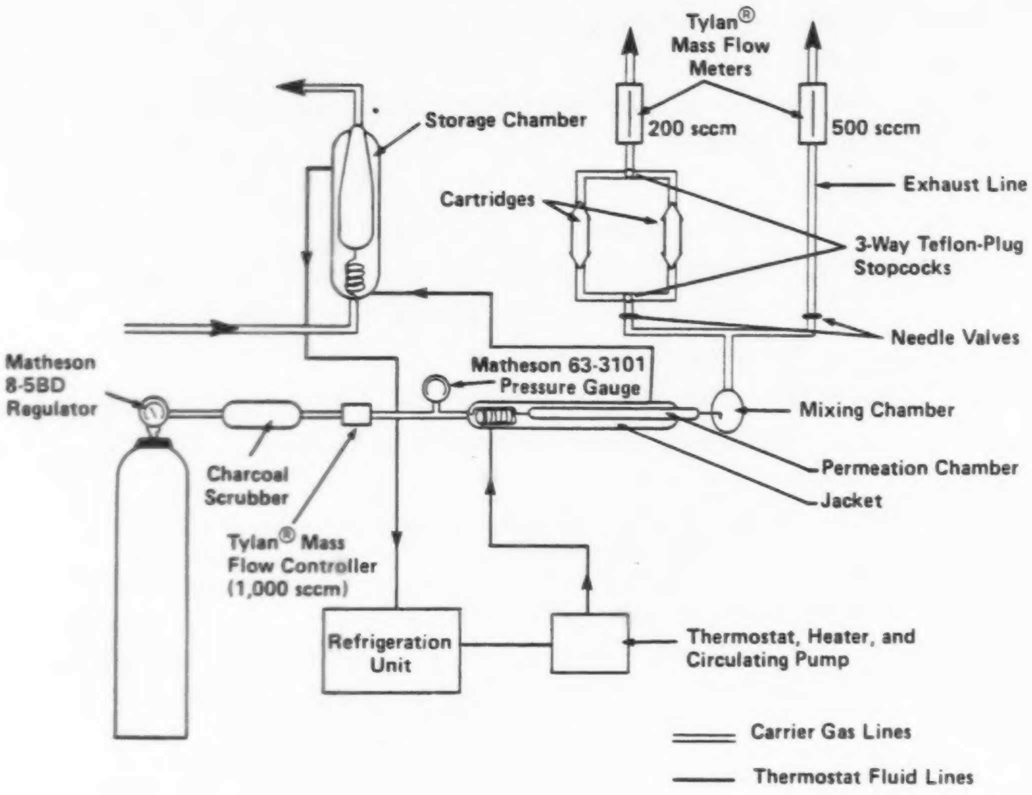
Tylan®
Mass Flow
Meters
Storage Chamber
200 sccm
500 sccm
Exhaust Line
Cartridges
3-Way Teflon-Plug
Stopcocks
Needle Valves
Matheson
8-5BD
Regulator
Matheson 63-3101
Pressure Gauge
Mixing Chamber
Permeation Chamber
Jacket
Charcoal
Scrubber
Tylan® Mass
Flow Controller
(1,000 sccm)
Refrigeration
Unit
Thermostat, Heater, and
Circulating Pump
Carrier Gas Lines
Thermostat Fluid Lines

Figure 1. Permeation system for generating N_2/vapor mixtures.

loaded; 4) load the Tenax cartridges; and 5) calculate the amounts loaded. In addition, this method provides quality assurance guidelines for the loading procedure.

3.0 PROCEDURE

3.1 Materials and Equipment

3.1.1 Permeation System (see Figure 1)
3.1.2 Nitrogen gas (99.995% purity)
3.1.3 Nylon gloves
3.1.4 Long glass hook (for retrieving permeation tubes)
3.1.5 Permeation tubes
3.1.6 Kimwipes®
3.1.7 Syringe, Pressure-Lok® gas type, 10 mL
3.1.8 Septum, Thermogreen™ LB-1 cylindrical, half hole type
3.1.9 Cleaning wires (for cleaning syringe needles)

3.2 Selection of the Permeation Tubes

3.2.1 Check the permeation data notebooks (located in the laboratory) to determine what permeation tubes are available for PFB and PFT.

NOTE: There is more than one permeation tube available for PFB and PFT. Each one has been assigned a unique number. The tubes may differ in their permeation rate and/or stability.

3.2.2 For each compound select one or two tubes whose total permeation rate is in the 4000-8000 ng/min range.

CAUTION

Consider only the permeation tubes whose permeation rates are stable.

NOTE: A permeation rate is considered stable when the mean rate has a coefficient of variation (CV) less than 10%. The mean permeation rate is calculated using the last five individual permeation rates.

3.2.3 In a bound RTI notebook, assigned for the project, list the number of selected permeation tubes for PFB and PFT, including their mean permeation rates.

3.3 Transfer of the Permeation Tubes to the Loading Chamber

3.3.1 Turn off the nitrogen flow in the storage chambers (see Figure 2).

3.3.2 Locate the chambers in which the selected permeation tubes are stored.

NOTE: Permeation tubes are stored in numerical order in the chambers. The range of numbers contained therein is listed outside each chamber.

WARNING

Open one storage chamber at a time. They contain toxic materials that are dangerous to your health.

3.3.3 Open the loading chamber as follows:

3.3.3.1 Lower the jack which supports the chamber.

3.3.3.2 Remove the clamp securing the two chamber sections.

3.3.3.3 Carefully open the loading chamber by moving the downstream section to the rear. Support it in the back with the jack.

3.3.3.4 Remove the "O" ring.

WARNING

Failure to wear nylon gloves when handling permeation tubes may cause skin damage and/or contamination of the permeation tubes.

3.3.4 With a long glass hook, remove one tube at a time and transfer immediately to the loading chamber.

WARNING

Perform this operation as quickly as possible. PFB and PFT are toxic compounds.

3.3.5 Reposition the "O" ring and reconnect the two sections of the loading chamber.

3.3.6 Attach the clamp.

3.3.7 Close and secure the storage chambers. Initiate nitrogen flow by opening the needle valve connected to the "house" nitrogen line.

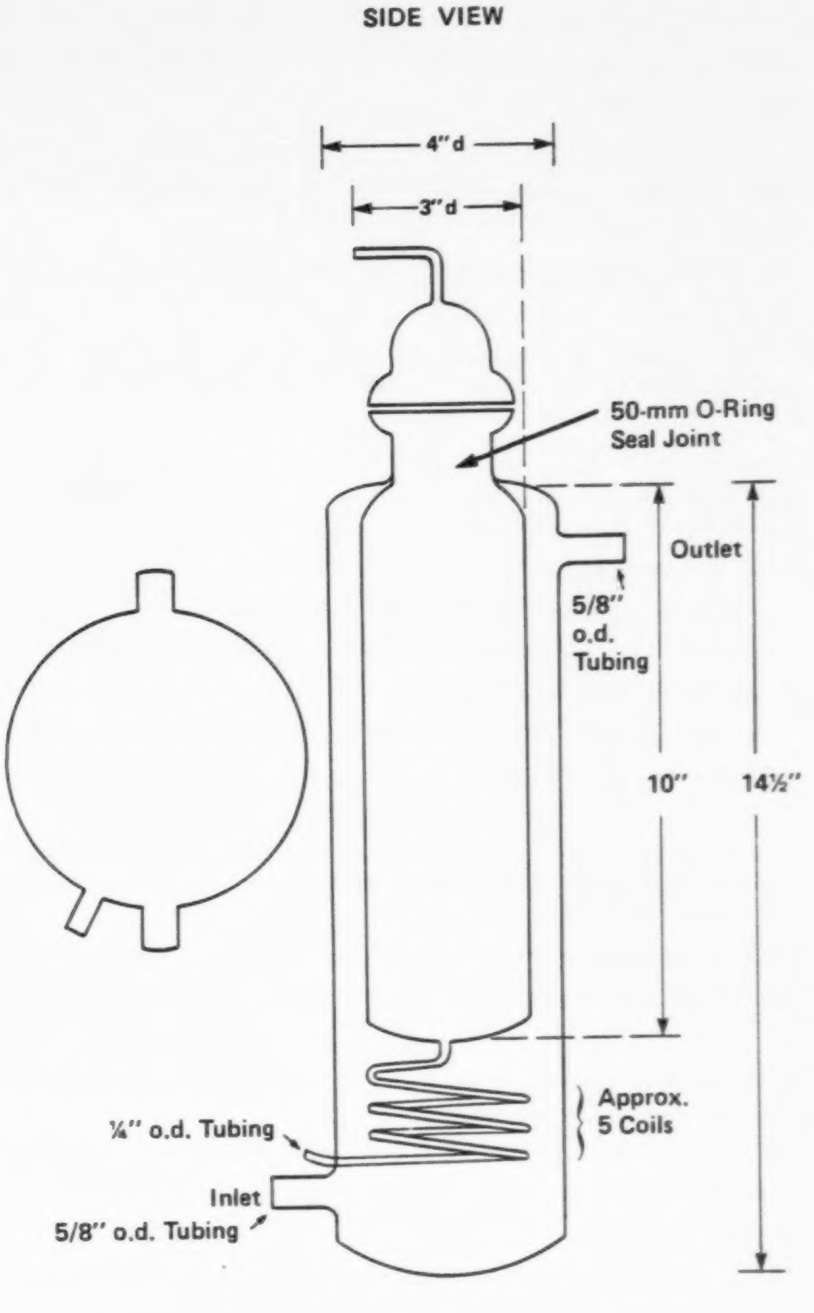
SIDE VIEW
4"d
3"d
50-mm O-Ring Seal Joint
Outlet
5/8" o.d. Tubing
10"
14½"
Approx. 5 Coils
¼" o.d. Tubing
Inlet
5/8" o.d. Tubing

Figure 2. Storage chamber.

CAUTION

Ensure that the rotameter indicates sufficient flow rate to distribute approximately 500 mL/min through each storage chamber.

3.4 Turning on the Permeation System

3.4.1 Turn on the read-out box.

NOTE: The read-out box displays the measurements of the mass flow meters and the mass flow controller. The labels on the box indicate the proper channels.

CAUTION

Ensure that the pressure regulator on the nitrogen tank is fully open counterclockwise (i.e., no pressure).

3.4.2 Turn on the nitrogen gas.

CAUTION

Ensure that the read-out box power is on before any nitrogen circulates through the system, otherwise the controller will be fully open and the excessive nitrogen flow may damage the system.

3.4.3 Carefully, increase the nitrogen pressure to the permeation system by rotating the pressure regulator T-handle clockwise, observing the gauge in the system at all times.

CAUTION

Do not allow the pressure to exceed 10 inches of water.

3.4.4 Set the outlet pressure at the nitrogen cylinder to 20 psi.

3.5 Setting the Flow Rates for the Loading

3.5.1 Rotate the two stopcocks to direct the nitrogen flow to the side with the loading tube (see Figure 1).

3.5.2 Turn the knob on the read-out box to the mass flow controller channel.

3.5.3 Adjust the total flow rate to ~360 mL/min with the mass flow controller adjustment knob.

3.5.4 Stabilize the flow rates for approximately 2 minutes.

3.5.5 Turn the knob to the cartridge flow rate channel. Adjust the nitrogen flow rate through the cartridge to ~85 mL/min with the proper needle valve.

3.5.6 Turn the knob to the exhaust flow rate channel.

3.5.7 Read the exhaust flow rate.

NOTE: The exhaust flow rate is equal to the total flow rate minus the flow rate through the cartridge.

CAUTION

If the total flow rate does not equal the sum of the exhaust flow rate and the cartridge flow rate, a gas leak should be suspected. Check the septum on the injection port (see Section 3.8.24 of this SOP to replace it). Leaks can also occur at the "O" ring which connects the two parts of the loading chamber.

3.6 Equilibration of the System

3.6.1 Allow the system to equilibrate for 90 minutes before starting the loading.

3.6.2 Reread the flow rates on the various channels before starting the loading.

3.7 Preparation of Tenax Cartridges Loaded with PFB/PFT

3.7.1 During the equilibration period, locate the cans in the freezer containing the cartridges to be loaded (see SOP-481-001, Storage of Tenax Cartridges).

3.7.2 Locate the Chain-of-Custody sheets (COC sheets) corresponding to the cans containing the cartridges to be loaded.

3.7.3 Allow the cans to reach room temperature before opening.

3.7.4 Open the cans and match each cartridge with the corresponding COC sheet.

3.7.5 Sign each COC sheet as received.

3.7.6 Contact the person in charge of logging-in the samples if any discrepancy is found between the samples and the COC sheets.

3.7.7 When the system is fully equilibrated, remove the cartridges to be loaded from the can.

3.7.8 Organize the cartridges in a test tube rack.

3.7.9 If field samples are to be loaded, organize them by sample sets (see NOTE below) and, if possible, in order of increasing study participant number. Include one QC set for everyone ten field samples.

NOTE: A sample set consists of all samples taken for a given participant for a specific matrix. (This includes duplicates but not quality assurance (QA) samples). QC sets differ according to the matrix. An air QC set consists of one blank and two spiked cartridges. A breath QC set consists of one blank, one spirometer blank and two spiked cartridges.

3.7.10 Seal the cartridges that will not be loaded in a properly labelled paint can and return the can to the freezer.

3.8 Loading of the Tenax Cartridges

CAUTION

Prior to loading the field samples or RMR cartridges, ensure that the system is working properly. Load a blank cartridge, following the loading instructions (see Steps 3.8.1-3.8.18) and analyze it as indicated in the QA section of this Standard Operating Procedure.

3.8.1 Prior to loading, clean the needle of the gas syringe with a thin wire.

CAUTION

Examine the syringe needle for burrs. Remove any found by gently "wiping" the needle, lengthwise, along No. 600 emory paper.

3.8.2 Turn the knob on the read-out box to the cartridge flow rate channel. Read the flow rate.

3.8.3 Holding the loading tube, carefully, insert the syringe needle through the septum.

3.8.4 After the flow rate has stabilized, slowly pull the plunger in the syringe half way back (approximately 5 mL).

3.8.5 Observe the change in the flow rate indicated on the read-out box.

CAUTION

If there is no change in the flow rate through the cartridge, remove the syringe and clean the needle again.

3.8.6 Wait until the flow rate stabilizes to the preset value.

3.8.7 Pull the plunger on the syringe an additional 5 mL to exactly 10.00 mL of volume.

3.8.8 Turn the knob on the read-out box to the exhaust flow rate channel.

3.8.9 Record the exhaust flow rate in the notebook and turn the knob back to the cartridge flow rate channel.

3.8.10 When the original flow rate though the cartridge is reached, close the valve in the syringe.

3.8.11 Record the flow rate through the cartridge in the notebook.

3.8.12 Holding the loading tube, withdraw the needle from the system.

3.8.13 Carefully rest the syringe on a clean surface.

3.8.14 Record the code of the sample to be loaded in the notebook.

3.8.15 Open the culture tube and hold the Tenax cartridge with a Kimwipe®.

WARNING

To avoid contamination, always handle the Tenax cartridges with Kimwipes® or nylon gloves.

3.8.16 Insert the needle in the back of the Tenax cartridge (see Figure 3).

WARNING

Perform all these operations as quickly as possible to minimize loss of standard.

3.8.17 Open the valve on the syringe and slowly inject the 10.00 mL of standard onto the Tenax cartridge.

3.8.18 Return the cartridge to its culture tube. Seal the tube.

3.8.19 Calculate the amount of external standards, loaded, using the following formula:

$$G = \frac{P \times V}{F_1 + F_2}$$

where: G = amount loaded (ng)

P = permeation rate (ng/min)

V = volume injected (mL)

F_1 = flow rate through the cartridge (mL/min)

F_2 = exhaust flow rate (mL/min)

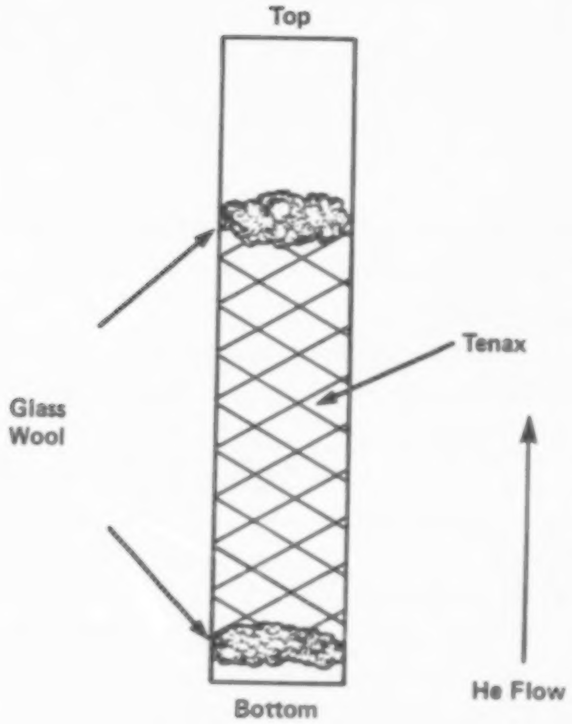
Top
Tenax
Glass Wool
He Flow
Bottom

Figure 3. Tenax cartridge.

3.8.20 Record the amount loaded in the notebook in the following table:

Sample Code	Cartridge Flow	Exhaust Flow	Total Flow	PFB ng	PFT ng

3.8.21 Sign the COC sheet (Figure 4) corresponding to the sample loaded and under operation performed enter the amounts of external standards loaded.

3.8.22 Store the loaded tube in a paint can.

3.8.23 To load another Tenax cartridge, follow instructions in Steps 3.8.1 to 3.8.23.

CAUTION

Change the septum every ten injections and at the end of the day.

3.8.24 Replace the septum as follows:

3.8.24.1 Turn the two stopcocks clockwise, simultaneously, to the side that is not in use.

3.8.24.2 Holding the loading tube, remove the septum carefully with a pair of tweezers.

3.8.24.3 Insert a new septum.

3.8.24.4 Divert the gas flow to the side with the loading tube by simultaneously turning the two stopcocks clockwise.

3.8.24.5 Wait approximately two minutes before loading another cartridge.

3.9 Turning Off the Permeation System

3.9.1 Open the pressure regulator slowly (counterclockwise) until fully open.

3.9.2 When the gauge in the system indicates no pressure, open the loading chamber as indicated in Step 3.3.3.

CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute, Analytical Sciences Division
Chemistry and Life Sciences Group, Research Triangle Park, NC 27709

SAMPLE CODE 2190-34-RMR-03 5-28-83	**TENAX BATCH:**	**VOLUME COLLECTED:**
SAMPLE TYPE: Tenax	**TEMPERATURE (°F)**	**VOLUME ANALYZED:**

RELIN-QUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	Initials		Date of Preparation	RMR mixture from Permeation System
Initials	Your Initials		Date of Loading	PFB = Amount Loaded (ng) PFT = Amount Loaded (ng) TO MS
Your Initials				

NOTE: "TO MS" indicates that after the loading of external standards, the samples are taken to the Mass Spectrometry Laboratory for analysis.

Figure 4.

3.9.3 Turn off the nitrogen flow in the storage chambers.

WARNING

Failure to wear nylon gloves when handling permeation tubes may cause skin damage and/or contamination of the permeation tubes.

3.9.4 With a long glass hook, remove the permeation tubes from the loading chamber and immediately transfer to the proper storage chambers.

3.9.5 Close the storage chambers.

3.9.6 Initiate nitrogen flow by opening the needle valve connected to the "house" nitrogen line.

CAUTION

Ensure that the rotometer indicates sufficient flow rate to distribute approximately 500 mL/min through each storage chamber.

3.9.7 Reposition the "O" ring on the loading chamber and move the upper part of the system back to the normal position.

3.9.8 Attach the clamp.

3.9.9 Turn off the read-out box.

3.10 Delivery of the Tenax Cartridges to the Mass Spectrometry Laboratory

3.10.1 Make a photocopy of the COC sheet for each sample loaded and retain the copy in a three-ring notebook.

3.10.2 Deliver the sealed paint cans with the loaded Tenax cartridges to the Mass Spectrometry Laboratory for GC/MS/COMP analysis.

3.10.3 Transfer the COC sheets and cans to the person assigned to sample analysis.

3.11 Quality Assurance for the Loading of External Standards

3.11.1 Analyze a blank cartridge loaded with PFB and PFT using a Varian 3700 gas chromatograph equipped with a thermal desorption injection unit and a flame ionization detector interfaced to a Varian CDS 111 integrator.

3.11.2 Use the same GC conditions as indicated on SOP 320-001 (Preparation of Tenax).

3.11.3 Change the temperature program to: 30°C (2 min), 4°C/min to 110°.

3.11.4 Calculate the instrumental response factors for PFB and PFT using the following formula:

$$\text{Response Factor} = \frac{\text{Area Counts}}{\text{ng loaded}}$$

3.11.5 Compare the calculated response factors with historical response factors generated under identical conditions.

NOTE: The historical data is stored as the mean response factor and standard deviation for each compound. A response factor is considered out of control when it deviates from the mean response factor by more than three standard deviations (3σ). This data is stored in a bound RTI notebook, assigned only for the instrument used.

3.11.6 If a mean response factor does not exist, create one after loading three cartridges and calculate the mean response factors.

3.11.7 If any major alterations are made to the GC (i.e., replacement of the column; replacement of the thermal desorption/ injection unit; cleaning of the detector), create new mean response factors.

3.11.8 If analysis of the first cartridge, indicates the two standards are in control begin loading the samples.

3.11.9 If one or both are out of control, determine the source of the problem.

WARNING

Do not load sample cartridges until the out-of-control condition has been corrected.

3.11.9.1 Change the septum as indicated in Step 3.8.24 and ensure that the "O" ring is properly seated.

3.11.10 Load a second blank cartridge and analyze it.

3.11.11 If the second cartridge is in control, proceed with the loading.

3.11.12 If it is out-of-control, load and analyze a column performance evaluation (CPM) cartridge. See SOP 630-002 for instructions.

NOTE: The analysis of a CPM cartridge provides a check on instrument conditions.

3.11.13 If the CPM cartridge is in control, repeat the leak check on the permeation system.

WARNING

Positive results from a CPM analysis indicates a failure in the permeation system. Be sure the problem is solved before loading external standards in any sample for GC/MS/COMP.

3.11.14 If the CPM cartridge is out-of-control, troubleshoot the instrument before proceeding with the loading.

RTI/ACS-SOP-630-004
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Standard Operating Procedure for Loading Deuterated Standards on Tenax Cartridges Using a Permeation System

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Standard Operating Procedure for Loading Deuterated Standards on Tenax Cartridges Using a Permeation System

RTIACS-SOP-630-004

1.0 SCOPE AND APPLICATION

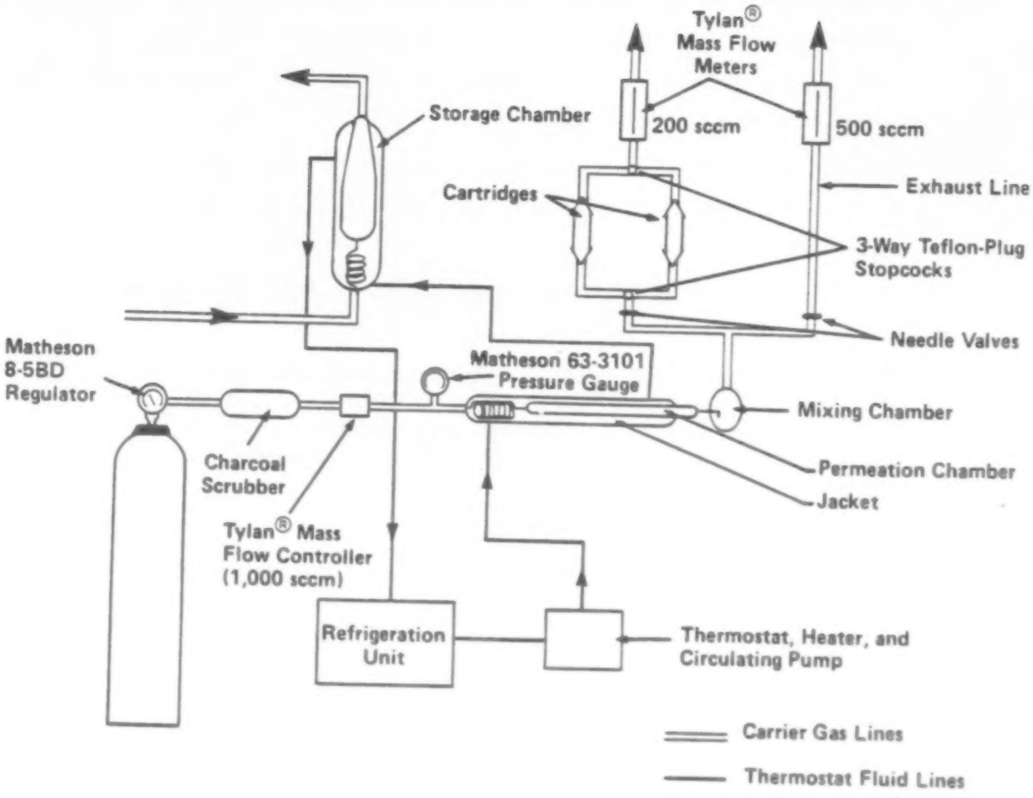
This Standard Operating Procedure describes the methodology for loading Tenax cartridges with deuterated standards via a permeation system. The Tenax cartridges loaded with deuterated standards are subsequently used for sampling. Sampling with these control cartridges allows for an assessment of premature breakthrough.

A permeation system consists of three main parts (see Figure 1): 1) a temperature-controlled chamber containing permeation tubes; 2) a mixing chamber; and 3) two cartridge holders enclosed in an insulated jacket. A stream of nitrogen flows through the system. The amounts of compounds transported downstream remain constant once the system has become equilibrated with the compounds to be loaded. The amounts of compounds can be determined by measuring the time and the gas flow through the cartridge.

This system may be used to load any volatile compound that will permeate at a constant rate under controlled conditions, and to inject a calibration standard onto a sorbent via syringe.

2.0 SUMMARY OF THE METHOD

The following routine shall be followed when Tenax cartridges are loaded with deuterated standards via a permeation system: 1) Determine the number of cartridges to be loaded; 2) Select the permeation tubes; 3) Determine the loading conditions to be used; 4) Equilibrate the system; 5) Load the cartridges; 6) Calculate the amounts of compounds loaded; 7) Ensure the integrity of the loading procedure; and 8) Pack and store the cartridges.



Tylan® Mass Flow Meters
200 sccm
500 sccm
Storage Chamber
Cartridges
Exhaust Line
3-Way Teflon-Plug Stopcocks
Needle Valves
Matheson 8-5BD Regulator
Matheson 63-3101 Pressure Gauge
Mixing Chamber
Charcoal Scrubber
Permeation Chamber
Jacket
Tylan® Mass Flow Controller (1,000 sccm)
Refrigeration Unit
Thermostat, Heater, and Circulating Pump
Carrier Gas Lines
Thermostat Fluid Lines

Figure 1. Permeation system for generating N_2/vapor mixtures.

3.0 PROCEDURE

3.1 Determination of the Number of Cartridges to Be Loaded

3.1.1 Obtain a copy of the field sampling schedule from the QA Officer.

3.1.2 Count the number of deuterated standard cartridges required for each matrix.

3.1.3 Send a memo to the person in charge of preparing the Tenax indicating by matrix the total number of deuterated cartridges needed (i.e., breath, personal air, fixed site).

3.2 Selection of the Permeation Tubes

3.2.1 Check the permeation notebooks (located in the laboratory) to see which permeation tubes are available for deuterated standards.

3.2.2 Select only the permeation tubes whose permeation rates are stable.

NOTE: A permeation rate is considered stable when the mean permeation rate has a coefficient of variation (CV) of less than 10%. The mean permeation rate is calculated using the last five individual permeation rates.

CAUTION

Do not use permeation tubes with permeation rates below 100 ng/min or above 1 x 10^5 ng/min.

3.2.3 In a bound RTI notebook assigned for the specific project, prepare a table including: 1) the numbers of the tubes to be used; 2) the names of the compounds; and 3) the corresponding mean permeation rates.

Tube #	Compound	Permeation Rate mean ± SD (CV)

3.3 Determination of the Permeation System Conditions

3.3.1 General Formula

For any compound, calculate the amounts loaded onto a cartridge using the following formula:

$$G = (P)(t)\left(\frac{F_1}{F_1 + F_2}\right)$$

Where:

G = amount loaded (ng)
P = permeation rate (ng/min)
t = time (min)
F_1 = flow rate through the cartridge (mL/min)
F_2 = exhaust flow rate (mL/min)

NOTE: The four variables G, t, F_1 and F_2 determine the loading conditions. Any three may be fixed and the fourth one calculated from the equation.

CAUTION

The following restrictions must be followed to minimize error: 1) Do not load for less than two minutes; 2) Do not load with a cartridge flow below 50 mL/min or above 150 mL/min; 3) Do not operate the system with a total flow below 250 mL/min.

3.3.2 Calculations

See Examples 1, 2 and 3 for loading specific amounts of compounds within a given mass range.

Example 1. Objective: to load benzene, chlorobenzene and chloroform onto a cartridge in the range of 200-500 ng per analyte.

Compound	Permeation Rate (ng/min)
Benzene	360
Chlorobenzene	270
Chloroform	520

At a fixed time and gas flow rate through the cartridge, compute the exhaust gas flow rate using the compound with the lowest permeation rate (chlorobenzene).

G = 200 ng   Fixing F_1 at 80 mL/min
P = 270 ng/min   t to 4 min,

Calculate:

$$G = (P)\ (t) \left(\frac{F_1}{F_1 + F_2} \right)$$

$$F_2 = \frac{(P)(t)(F_1)}{G} - F_1$$

$$F_2 = \frac{(270\ \text{ng/min})(4\ \text{min})(80\ \text{mL/min})}{200\ \text{ng}} - 80\ \text{mL/min}$$

$$F_2 = 352\ \text{mL/min}$$

Using these conditions, calculate the amount of compound loaded for the tube with the highest permeation rate (chloroform).

G = ?
P = 520 ng/min
F_1 = 80 mL/min
F_2 = 352 mL/min
t = 4 min

$$G = (P)(t) \left(\frac{F_1}{F_1 + F_2} \right)$$

$$G = (520\ \text{ng/min})(4\ \text{min}) \left(\frac{80\ \text{mL/min}}{80\ \text{mL/min} + 352\ \text{mL/min}} \right)$$

$$G = 385\ \text{ng}$$

NOTE: All the values obtained are within the acceptable range.

EXAMPLE 2: Objective: To load benzene, chlorobenzene, and chloroform onto a cartridge in the 100-200 ng per analyte range.

Compound	Permeation Rate (ng/min)
Benzene	360
Chlorobenzene	270
Chloroform	520

Follow the same procedure as in Example 1: 1) consider 100 ng of chlorobenzene (lowest permeation rate); 2) fix the time and flow through the cartridge; and 3) calculate the exhaust flow rate.

G = 100 ng

P = 270 ng/min

F_1 = 80 ml/min

t = 4 min

F_2 = ?

$$G = (P)(t)\left(\frac{F_1}{F_1 + F_2}\right)$$

$$F_2 = \frac{(P)(t)(F_1)}{G} - F_1$$

$$F_2 = \frac{(270\ \text{ng/min})(4\text{min})(80\ \text{mL/min})}{100\ \text{ng}} - 80\ \text{mL/min}$$

$$F_2 = 784\ \text{mL/min}$$

This exhaust flow rate is higher than the maximum acceptable value (see Section 3.2.1).

Change the values to:

t = 2.5 min

F_1 = 60 ml/min

F_2 = ?

$$F_2 = \frac{(P)(t)(F_1)}{G} - F_1$$

$$F_2 = \frac{(270\ \text{ng/min})(2.5\ \text{min})(60\ \text{mL/min})}{100\ \text{ng}} - 60\ \text{mL/min}$$

$$F_2 = 345\ \text{mL/min}$$

NOTE: These conditions are acceptable. Now, calculate the amount of compound loaded for the tube with the highest permeation rate (chloroform).

G = ?

P = 520 ng/min

F_1 = 60 mL/min

F_2 = 345 mL/min

t = 2.5 min

$$G = (P)(t)\left(\frac{F_1}{F_1 + F_2}\right)$$

$$G = (520\ \text{ng/min})(2.5\ \text{min})\left(\frac{60\ \text{mL/min}}{60\ \text{mL/min} + 345\ \text{mL/min}}\right)$$

$$G = 193\ \text{ng}$$

NOTE: All the values obtained are within the acceptable range.

EXAMPLE 3: Objective: To load benzene, chlorobenzene and chloroform onto a cartridge in the range of 500-1000 ng per analyte.

Follow the same procedure as in Examples 1 and 2. Consider 500 ng of chlorobenzene.

$G = 500$ ng $\qquad G = (P)(t)\left(\frac{F_1}{F_1 + F_2}\right)$

$P = 270$ ng/min $\qquad F_2 = \frac{(P)(t)(F_1)}{G} - F_1$

$F_1 = 80$ ml/min $\qquad F_2 = \frac{(270 \text{ ng/min})(4 \text{ min})(80 \text{ mL/min})}{500 \text{ ng}} - 80 \text{ mL/min}$

$F_2 = ?$ $\qquad F_2 = 93$ mL/min

$t = 4$ min

The total flow rate (173 mL/min) is below the acceptable level (see 3.2.1 CAUTION). Alternatively, calculate the total flow rate for twice the loading time.

$G = 500$ ng $\qquad F_2 = \frac{(P)(t)(F_1)}{G} - F_1$

$P = 270$ ng/min $\qquad F_2 = \frac{(270 \text{ ng/min})(8 \text{ min})(80 \text{ mL/min})}{500 \text{ ng}} - 80 \text{ mL/min}$

$F_1 = 80$ mL/min $\qquad F_2 = 266$ mL/min

$g = 8$ min

$F_2 = ?$

NOTE: This exhaust rate is acceptable.

Finally, calculate the amount of compound loaded for the tube with the highest permeation rate.

$G = ?$

$$G = (P)(t)\left(\frac{F_1}{F_1 + F_2}\right)$$

$P = 520$ ng/min

$$G = (520\ \text{ng/min})(8\ \text{min})\left(\frac{80\ \text{mL/min}}{80\ \text{mL/min} + 266\ \text{mL/min}}\right)$$

$F_1 = 80$ mL/min

$$G = 962\ \text{ng}$$

$g = 8$ min

$F_2 = 266$ mL/min

NOTE: All the values obtained are within the acceptable range.

3.4 Materials and Equipment

3.4.1 Permeation system (see Figure 1)

3.4.2 Nitrogen gas (99.995% purity)

3.4.3 Nylon gloves

3.4.4 Long glass hook (for retrieving permeation tubes)

3.4.5 Permeation tubes

3.4.6 Kimwipes®

3.4.7 Stopwatch

3.5 Equilibration of the Permeation System

3.5.1 Turn off the nitrogen flow in the storage chambers (see Figure 2).

3.5.2 Locate the chambers in which the selected permeation tubes are stored.

NOTE: Permeation tubes are stored in numerical order in the chambers. The range of numbers contained therein is listed outside each chamber.

WARNING

Open one storage chamber at a time. They contain toxic materials that are dangerous to your health.

3.5.3 Open the loading chamber as follows:

3.5.3.1 Lower the jack which supports the chamber.

3.5.3.2 Remove the clamp securing the two chamber sections.

3.5.3.3 Carefully open the loading chamber by moving the downstream section to the rear.

3.5.3.4 Remove the "O" ring.

WARNING

Failure to wear nylon gloves when handling permeation tubes may result in skin damage and/or contamination of the permeation tubes.

3.5.4 With a long glass hook, remove the selected permeation tubes from the storage chamber and transfer immediately to the loading chamber.

WARNING

Perform this operation as quickly as possible. The tubes contain toxic materials, some of which are cancer suspect agents.

3.5.5 After the last permeation tube has been transferred, reposition the "O" ring and reconnect the two sections of the loading chamber.

3.5.6 Attach the clamp.

3.5.7 Close and secure the storage chambers. Initiate nitrogen flow by opening the needle valve connected to the "house" nitrogen line.

CAUTION

Ensure that the rotometer indicates sufficient flow rate to distribute approximately 500 mL/min through each storage chamber.

3.5.8 Turn on the read-out box.

NOTE: The read-out box displays the measurements of the mass flow meters and the mass flow controller. The labels on the box indicate the proper channels.

3.5.9 Ensure that the pressure regulator on the nitrogen tank is fully open (counterclockwise) (i.e., no pressure).

3.5.10 Turn on the nitrogen gas.

CAUTION

Ensure that the read-out box power is on before any nitrogen flows through the system, otherwise the controller will be fully open and the excessive nitrogen flow may damage the system.

3.5.11 Carefully increase the nitrogen pressure to the permeation system by rotating the pressure regulator T-handle clockwise, observing the gauge in the system at all times. Do not allow the pressure to exceed 10 inches of water.

3.5.12 Set the outlet pressure at the nitrogen cylinder to 20 psi.

3.5.13 Rotate the two stopcocks to direct the nitrogen flow to the side where the Tenax cartridge will be loaded. (see Figure 1).

NOTE: When a cartridge is not being loaded, a dummy cartridge is placed in the loading position.

3.5.14 Turn the knob on the read-out box to the total flow rate channel. Adjust the total flow rate with the appropriate controller adjustment knob.

3.5.15 Stabilize the flow rates for approximately two minutes.

3.5.16 Turn the knob to the cartridge flow rate channel. Adjust the nitrogen flow rate through the cartridge with the proper needle valve.

3.5.17 Turn the knob to the exhaust flow rate channel.

3.5.18 Read the exhaust flow rate.

NOTE: The exhaust flow rate is equal to the total flow rate minus the flow rate through the cartridge.

CAUTION

If the total flow rate does not equal the sum of the exhaust flow rate and the cartridge flow rate, a gas leak should be suspected. Ensure that the "O" ring is properly seated and the clamp is secure.

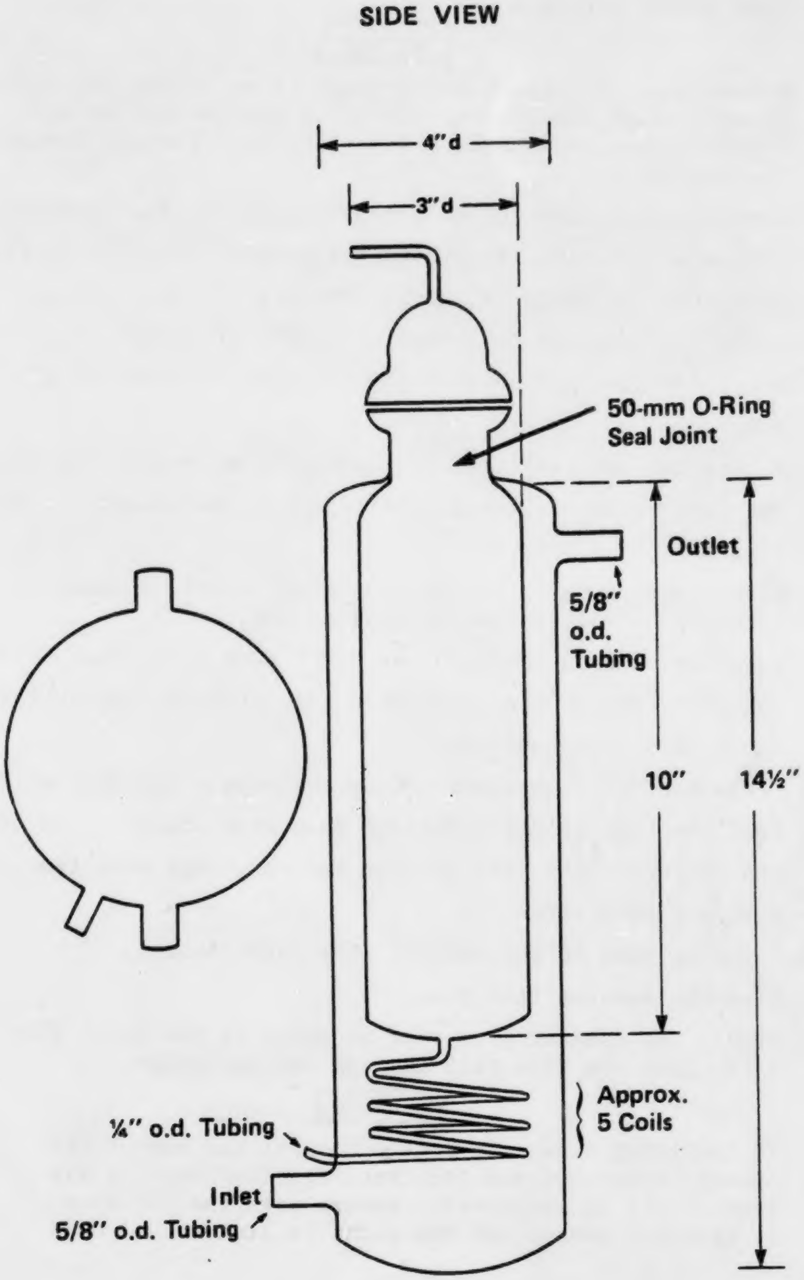

SIDE VIEW
4"d
3"d
50-mm O-Ring Seal Joint
Outlet
5/8" o.d. Tubing
10"
14½"
Approx. 5 Coils
¼" o.d. Tubing
Inlet
5/8" o.d. Tubing

Figure 2. Storage chamber.

3.5.19 Allow the system to equilibrate for 90 minutes before loading cartridges.

3.5.20 Recheck loading conditions. Read again the flow rates on the different channels.

3.6 Loading of the Tenax Cartridges

CAUTION

Be sure the background of the Tenax cartridges is acceptable before loading them. Follow QA section of SOP-320-001.

3.6.1 Divert the nitrogen flow to the side that will not be used for loading cartridges (see Figure 1) by turning the two stopcocks clockwise, simultaneously.

3.6.2 Loosen the Cajon® fittings and insert the Tenax cartridge with the bottom part down (see Figure 3).

WARNING

Use Kimwipes® when handling the cartridge to avoid contamination.

3.6.3 Tighten the bottom fitting first, then the upper one.

3.6.4 Rotate the two stopcocks clockwise, simultaneously, to direct the gas flow through the cartridge.

3.6.5 Concurrently, start the stopwatch.

3.6.6 Turn the knob on the read-out box to the cartridge flow rate channel. Record this flow rate in the notebook.

3.6.7 Turn the knob to the channel displaying the exhaust flow rate. Record this flow rate in the notebook.

3.6.8 Calculate the time needed to load the amounts desired as follows:

$$t = \frac{G}{P} \times \left(\frac{F_1 + F_2}{F_1} \right)$$

(see Section 3.2.1 for definitions). Do the calculation based only on the compound whose permeation tube has the highest or lowest permeation rate.

3.6.9 At the calculated time rotate the two stopcocks to direct gas flow away from the cartridge being loaded.

3.6.10 Handling the cartridge with a Kimwipe®, loosen the Cajon® fittings, the bottom side first.

3.6.11 Remove the cartridge and return it to its culture tube. Seal the tube.

3.6.12 Label the tube. Include the following information: 1) project number; 2) deuterated standard mixture followed by a number indicating the order of loading; and 3) the date. For example:

2190-20-Deut. Standards-02.
5-25-83

3.6.13 Prepare a Sampling Protocol/Chain-of-Custody sheet with the information on the Chain of Custody (COC) side as shown in Figure 3.

CAUTION

Do not make an entry in the SAMPLE CODE box. This area will be completed in the field.

3.6.14 Analyze by GC/FID the first cartridge loaded, according to the conditions specified in the QA section of SOP-320-001 (Preparation of Tenax).

3.6.15 To load another cartridge follow instructions in Steps 3.6.2 to 3.6.13.

3.7 Turning Off the Permeation System

3.7.1 Open the pressure regulator slowly (counterclockwise) until fully open.

3.7.2 When the gauge in the system indicates no pressure, open the loading chamber as follows: 1) lower the jack; 2) remove the clamp; 3) carefully move the upper part of the system back; 4) use the jack to support the upper part of the system; and 5) remove the "O" ring.

3.7.3 Turn off the nitrogen flow in the storage chambers.

WARNING

Failure to wear nylon gloves when handling permeation tubes may cause skin damage and/or contamination of the permeation tubes.

CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute, Analytical Sciences Division
Chemistry and Life Sciences Group, Research Triangle Park, NC 27709

SAMPLE CODE	TENAX BATCH:	VOLUME COLLECTED:
SAMPLE TYPE:	TEMPERATURE (°F)	VOLUME ANALYZED:

RELIN-QUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED

Figure 3. Chain-of-custody record.

3.7.4 With a long glass hook remove one tube at a time and immediately transfer to the respective storage chamber.

3.7.5 After the last tube has been transferred close the storage chambers.

3.7.6 With the needle valve that is connected to the house nitrogen line, turn the nitrogen gas on.

3.7.7 Reposition the "O" ring on the loading chamber and move the upper part of the system back to the normal position.

3.7.8 Attach the clamp.

3.7.9 Turn off the read-out box.

3.8 Storage of the Deuterated Standard Cartridges

3.8.1 Secure the cartridge inside the Kimax® tube with a glass wool plug to avoid breakage during transport.

3.8.2 Label the top of the screw cap with the following symbol: D*.

NOTE: * The star indicates that deuterated standards have been loaded onto the cartridge. This symbol will also be added to the participant's code.

3.8.3 Store the cartridges in a sealed paint can in the freezer until they are ready to be sent to the field.

3.9 Calculations of the Amounts Loaded

3.9.1 For every compound, calculate the amount loaded onto a cartridge using the general formula:

$$G = (P)(t)\left(\frac{F_1}{F_1 + F_2}\right)$$

(See Section 3.2.1 for definitions).

3.9.2 Do all the calculations in the RTI notebook.

3.10 Quality Assurance of the Deuterated Standard Cartridges

After a group of cartridges have been loaded with the deuterated standard compounds, one or two shall be analyzed by GC/FID according to the conditions specified in the QA Section of SOP-320-001 (Preparation of Tenax).

RTI/ACS-SOP-630-005
May 1984
Revision 0

Standard Operating Procedure for Preparing Response Factor Tenax Cartridges Using a Permeation System

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6/4/84
QA Officer, RTI/ACS Date

E Pellizzari 6/4/84
Vice-President, RTI/ACS Date

Standard Operating Procedure for Preparing Response Factor Tenax Cartridges Using a Permeation System

RTI/ACS-SOP-630-005

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure describes the methodology for loading Tenax cartridges via a dynamic dilution system, hereafter referred to as the "Permeation System" (see Figure 1). A permeation system consists of three main parts: (1) a temperature-controlled chamber containing permeation tubes; (2) a mixing chamber; and (3) two cartridge holders enclosed in an insulated jacket. A stream of nitrogen circulates through the system. The amounts of compounds transported downstream remain constant once the walls of the system become saturated. The amounts of compounds can be determined by measuring the time and the gas flow through the cartridge. One of the most important applications of this system is loading known quantities of compounds onto Tenax cartridges. These cartridges are used to determine the Response Factor (RF) of the GC/MS/COMP system. The samples prepared for this purpose are designated as RF cartridges.

This system may be applied to any volatile compound that may be prepared as a permeation tube with a constant permeation rate under controlled conditions. The permeation system may be used to load known quantities of compounds not only onto Tenax, but onto any other sorbent (i.e., charcoal). In addition, with minor modifications this system may be used to load external standards onto a field sample via syringe.

2.0 SUMMARY OF THE METHOD

The following routine shall be followed when Tenax cartridges are loaded with RF compounds via a permeation system: (1) select the appropriate permeation tubes; (2) determine the loading conditions to be used (i.e., flow rates and duration of loading); (3) equilibrate the system; (4) check the background of the Tenax cartridges; (5) load the

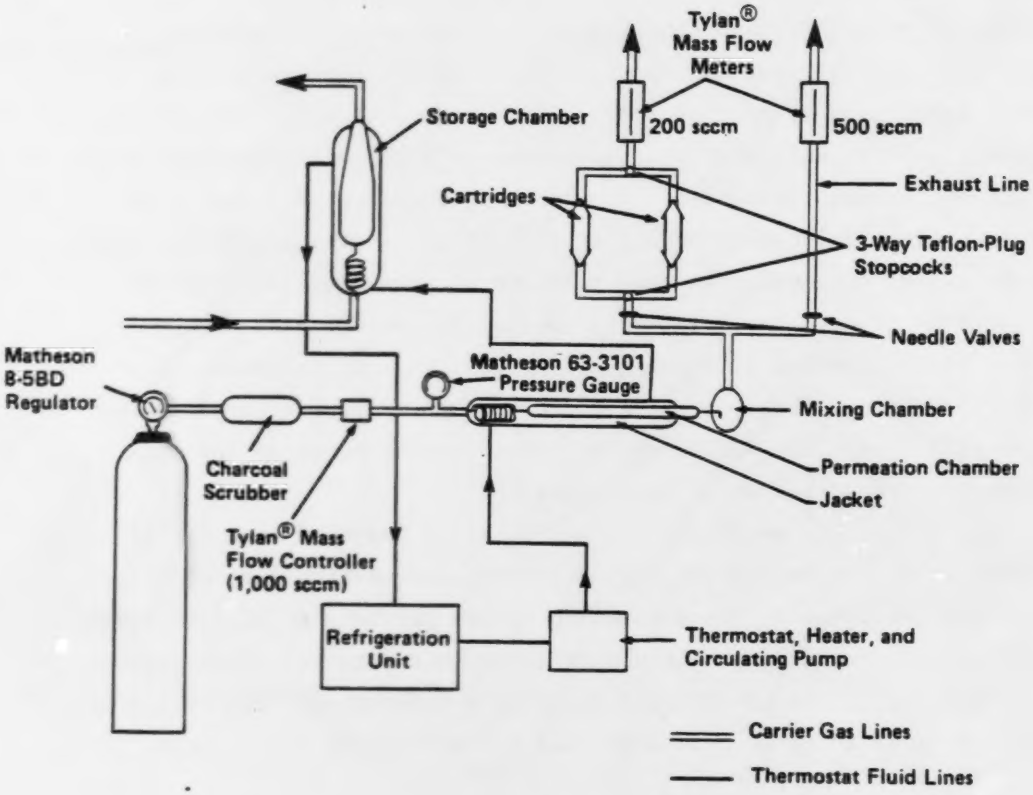
Tylan®
Mass Flow
Meters
Storage Chamber
200 sccm
500 sccm
Exhaust Line
Cartridges
3-Way Teflon-Plug
Stopcocks
Needle Valves
Matheson
B-5BD
Regulator
Matheson 63-3101
Pressure Gauge
Mixing Chamber
Charcoal
Scrubber
Permeation Chamber
Jacket
Tylan® Mass
Flow Controller
(1,000 sccm)
Refrigeration
Unit
Thermostat, Heater, and
Circulating Pump
Carrier Gas Lines
Thermostat Fluid Lines

Figure 1. Permeation system for generating N_2/vapor mixtures.

cartridges; (6) calculate the amounts of compounds loaded; (7) assure the integrity of the loading procedure; and (7) add the external standards. See Appendix I of this SOP for determining permeation rates for the permeation tubes.

3.0 PROCEDURE

3.1 Selection of Permeation Tubes

3.1.1 In a bound RTI notebook, assigned for a specific project, list all the compounds to be loaded.

3.1.2 Check the permeation notebooks (located in the laboratory) to see what permeation tubes are available for each of the target compounds.

NOTE: There is more than one permeation tube available for almost every compound. Each one has been assigned a unique number. The tubes may differ in their permeation rate and/or stability.

3.1.3 For each target compound select the most stable permeation tube whose permeation rate is nearest to ~300 ng/min.

3.1.4 Complete the table below in the RTI notebook, entering the number of the tube to be used and the corresponding permeation rate.

Tube #	Compound	Permeation Rate Mean ± S.D. (CV)

NOTE: The mean permeation rate is calculated using the last five individual permeation rates.

3.2 Determination of the Permeation System Conditions

3.2.1 General Formula

For any compound, calculate the amount loaded onto a cartridge using the following formula:

$$G = (P)\ (t)\left(\frac{F_1}{F_1 + F_2}\right)$$

where: G = amount loaded (ng)
P = permeation rate (ng/min)
t = time (min)
F_1 = flow rate through the cartridge (mL/min)
F_2 = exhaust flow rate (mL/min)

NOTE: The four variables, G, t, F_1 and F_2, determine the loading conditions. Any three of them may be fixed and the fourth one calculated from the equation.

CAUTION

Do not use permeation tubes with permeation rates below 100 ng/min. Do not use permeation tubes with permeation rates higher than 1 x 10^5 ng/min. Do not load a cartridge using a cartridge flow below 50 mL/min or above 150 mL/min. Do not operate the system with a total flow below 250 mL/min or above 600 mL/min. Do not sample for less than two minutes. These restrictions decrease the possibility of error.

3.2.2 Calculations

See Examples 1, 2 and 3 for loading specific amounts of compounds within a given range.

Example 1. Objective: To load benzene, chlorobenzene and chloroform onto a cartridge in the range of 200-500 ng per analyte.

Compound	Permeation Rate (ng/min)
benzene	360
chlorobenzene	270
chloroform	520

At a fixed time and gas flow rate through the cartridge, compute the exhaust gas flow rate using the compound with the lowest permeation rate (chlorobenzene).

G = 200 ng Fixing F_1 at 80 mL/min
P = 270 ng/min t to 4 min,

Calculate:

$$G = (P)\ (t) \left(\frac{F_1}{F_1 + F_2} \right)$$

$$F_2 = \frac{(P)\ (t)\ (F_1)}{G} - F_1$$

$$F = \frac{(270\ \text{ng/min})\ (4\ \text{min})\ (80\ \text{mL/min})}{200\ \text{ng}} - 80\ \text{mL/min}$$

$$F_2 = 352\ \text{mL/min}$$

Using these conditions calculate the amount of compound loaded for the tube with the highest permeation rate (chloroform).

$G = ?$
$P = 520$ ng/min
$F_1 = 80$ mL/min
$F_2 = 352$ mL/min
$t = 4$ min

$$G = (P)\ (t) \left(\frac{F_1}{F_1 + F_2} \right)$$

$$G = (520 \text{ ng/min})\ (4 \text{ min})\ \frac{80 \text{ mL/min}}{80 \text{ mL/min} + 352 \text{ mL/min}}$$

$G = 385$ ng

NOTE: All the values obtained are within the acceptable range.

Example 2. Objective: To load benzene, chlorobenzene and chloroform onto a cartridge in the 100-200 ng per analyte range.

Compound	Permeation Rate (ng/min)
benzene	360
chlorobenzene	270
chloroform	520

Follow the same procedure as in Example 1: (1) consider 100 ng of chlorobenzene (lowest permeation rate); (2) fix the time and flow through the cartridge; and (3) calculate the exhaust flow rate.

$G = 100$ ng
$P = 270$ ng/min
$F_1 = 80$ mL/min
$t = 4$ min
$F_2 = ?$

$$G = (P)\ (t) \left(\frac{F_1}{F_1 + F_2} \right)$$

$$F_2 = \frac{(P)\ (t)\ (F_1)}{G} - F_1$$

$$F_2 = \frac{(270 \text{ ng/min})\ (4 \text{ min})\ (80 \text{ mL/min})}{100 \text{ ng}} - 80 \text{ mL/min}$$

$F_2 = 784$ mL/min

This exhaust flow rate is higher than the maximum acceptable value (see Section 3.2.1).

t = 2.5 min
F = 60 mL/min
F_2 = ?

$$F_2 = \frac{(P)\ (t)\ ^{(F_1)}}{G} - F_1.$$

$$F_2 = \frac{(270 \text{ ng/min})\ (2.5 \text{ min}\ (60 \text{ mL/min})}{100 \text{ ng}} = 60 \text{ mL/min}$$

$$F_2 = 345 \text{ mL/min}$$

NOTE: These conditions are acceptable. Now, calculate the amount of compound loaded for the tube with the highest permeation rate (chloroform).

G = ?
P = 520 ng/min
F_1 = 60 mL/min
F_2 = 345 mL/min
g = 2.5 min

$$G = (P)\ (t) \left(\frac{F_1}{F_1 + F_2} \right)$$

$$G = (520 \text{ ng/min})\ (2.5 \text{ min})\ \frac{60 \text{ mL/min}}{60 \text{ mL/min} + 345 \text{ mL/min}}$$

$$G = 193 \text{ ng}$$

NOTE: All the values obtained are within the acceptable range.

Example 3. Objective: To load benzene, chlorobenzene and chloroform onto a cartridge in the range of 500-1000 ng per analyte.

Follow the same procedure as in Examples 1 and 2.

Consider 500 ng for chlorobenzene.

G = 500 ng
P = 270 ng/min
F_1 = 80 mL/min
F_2 = ?
t = 4 min

$$G = (P)\ (t) \left(\frac{F_1}{F_1 + F_2} \right)$$

$$F_2 = \frac{(P)\ (t)\ ^{(F_1)}}{G} - F_1$$

$$F_2 = \frac{(270 \text{ ng/min})\ (4 \text{ min})\ (80 \text{ mL/min})}{500 \text{ ng}} - 80 \text{ mL/min}$$

$$F_2 = 93 \text{ mL/min}$$

The total flow rate (173 mL/min) is below the acceptable level (see 3.2.1 CAUTION).

Now consider the same flow rate through the cartridge but for twice the length of time.

$G = 500$ ng
$P = 270$ (ng/min)
$F_1 = 80$ mL/min
$t = 8$ min
$F_2 = ?$

$$F_2 = \frac{(P)\ (t)\ (F_1)}{G} - F_1$$

$$F_2 = \frac{(270\ \text{ng/min})\ (8\ \text{min})\ (80\ \text{mL/min})}{500\ \text{ng}} - 80\ \text{mL/min}$$

$$F_2 = 266\ \text{mL/min}$$

NOTE: This exhaust rate is acceptable.

Finally, calculate the amount of compound loaded for the tube with the highest permeation rate.

$G = ?$
$P = 520$ (ng/min)
$F_1 = 80$ mL/min
$t = 8$ min
$F_2 = 266$ mL/min

$$G = (P)\ (t) \left(\frac{F_1}{F_1 + F_2}\right)$$

$$G = (520\ \text{ng/min})\ (8\ \text{min}) \left(\frac{80\ \text{mL/min}}{80\ \text{mL/min} + 266\ \text{mL/min}}\right)$$

$$G = 962\ \text{ng}$$

NOTE: All the values obtained are within the acceptable range.

3.3 Materials and Equipment

3.3.1 Permeation system (see Figure 1)
3.3.2 Nitrogen gas (99.995% purity)
3.3.3 Nylon gloves
3.3.4 Long glass hook (for retrieving permeation tubes)
3.3.5 Permeation tubes
3.3.6 Kimwipes®
3.3.7 Stopwatch

3.4 Equilibration of the Permeation System

3.4.1 Turn off the nitrogen flow in the storage chambers (see Figure 2).

3.4.2 Locate the chambers in which the selected permeation tubes are stored.

WARNING

Open one storage chamber at a time. They contain toxic materials that are dangerous to your health.

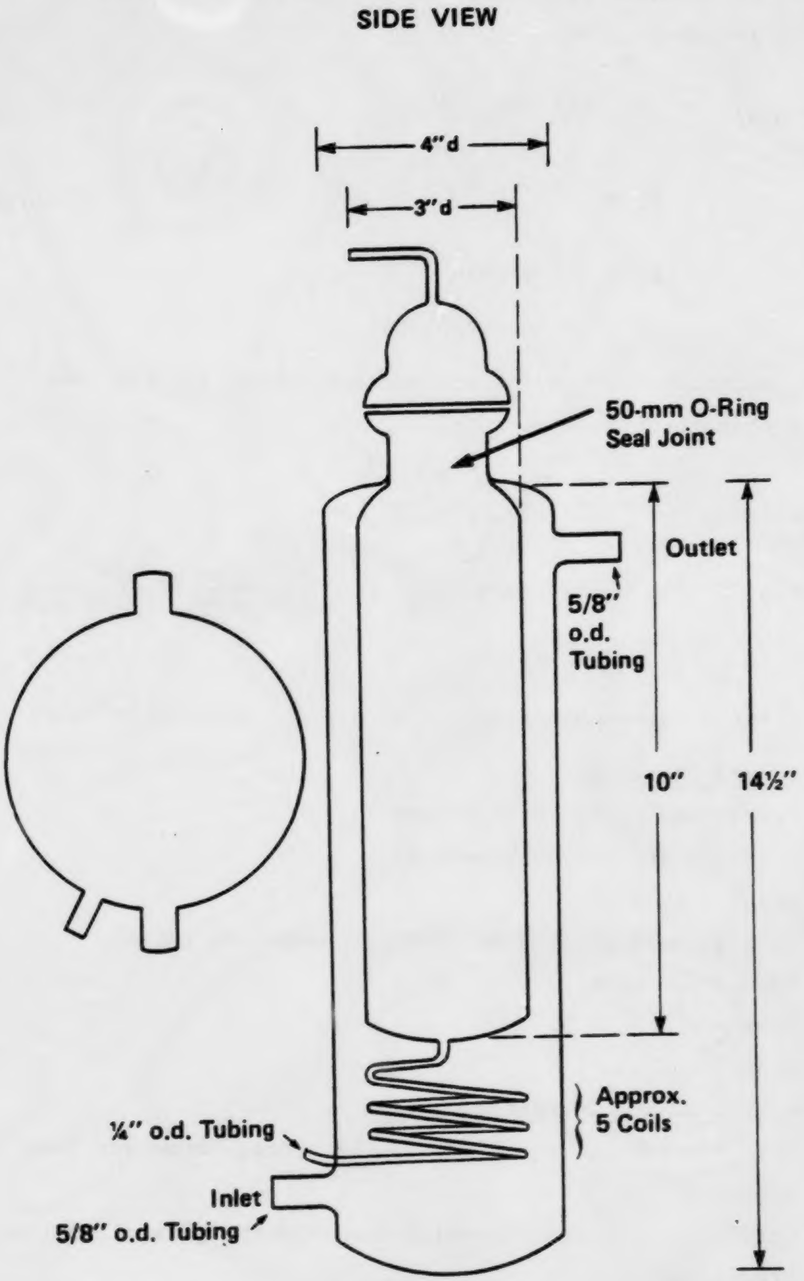
SIDE VIEW
4"d
3"d
50-mm O-Ring Seal Joint
Outlet
5/8" o.d. Tubing
10"
14½"
Approx. 5 Coils
¼" o.d. Tubing
Inlet
5/8" o.d. Tubing

Figure 2. Storage chamber.

3.4.3 Open the loading chamber as follows:

3.4.3.1 Lower the jack which supports the chamber.

3.4.3.2 Remove the clamp securing the two chamber sections.

3.4.3.3 Carefully open the loading chamber by moving the downstream section to the rear.

3.4.3.4 Use jack to support the section.

3.4.3.5 Remove the "O" ring.

WARNING

Failure to wear nylon gloves when handling permeation tubes may result in skin damage and/or contamination of the pereation tubes.

3.4.4 With a long glass hook, remove the selected permeation tubes from the storage chamber and transfer immediately to the loading chamber.

WARNING

Perform this operation as quickly as possible. The tubes contain toxic materials, some of which are cancer-suspect agents.

3.4.5 After the last permeation tube has been transferred, reposition the "O" ring and reconnect the two sections of the loading chamber.

3.4.6 Attach the clamp.

3.4.7 Close and secure the storage chambers. Initiate nitrogen flow by opening the needle valve connected to the "house" nitrogen line.

CAUTION

Ensure that the rotometer indicates sufficient flow rate to distribute approximately 500 mL/min through each storage chamber.

3.4.8 Turn on the read-out box.

NOTE: The read-out box displays the measurements of the mass flow meter and the mass flow controllers. The labels on the box indicate the proper channels.

3.4.9 Ensure that the pressure regulator on the nitrogen tank is fully open (counter-clockwise) (i.e., no pressure).

3.4.10 Turn on the nitrogen gas.

CAUTION

Ensure that the read-out box power is on before any nitrogen circulates through the system, otherwise, the controller will be fully open and the excessive nitrogen flow may damage the system.

3.4.11 Carefully, increase the nitrogen pressure to the permeation system by rotating the pressure regulator T-handle clockwise, observing the gauge in the system at all times. Do not allow the pressure to exceed 10 inches of water.

3.4.12 Set the outlet pressure at the nitrogen cylinder to 20 psi.

3.4.13 Rotate the two stopcocks to direct the nitrogen flow to the side where the Tenax cartridge will be loaded (see Figure 1).

NOTE: When a cartridge is not being loaded, a dummy cartridge is placed in the loading position.

3.4.14 Turn the knob on the read-out box to the total flow rate channel. Adjust the total flow rate with the appropriate controller adjustment knob.

3.4.15 Stabilize the flow rates for approximately two minutes.

3.4.16 Turn the knob to the cartridge flow rate channel. Adjust the nitrogen flow rate through the cartridge with the proper needle valve.

3.4.17 Turn the knob to the exhaust flow rate channel.

3.4.18 Read the exhaust flow rate.

NOTE: The exhaust flow rate is equal to the total flow rate minus the flow rate through the cartridge.

CAUTION

If the total flow rate does not equal the sum of the exhaust flow rate and the cartridge flow rate, a gas leak should be suspected. Ensure that the "O" ring is properly seated and the clamp is secure.

3.4.19 Allow the system to equilibrate for 90 minutes before loading any cartridges.

3.4.20 Recheck loading conditions. Read again the flow rates on the different channels.

3.5 Loading of the Tenax Cartridges

If all RF compounds will be loaded only via a permeation system, be sure the background of the Tenax cartridges is acceptable before loading them. Follow Background Check section of SOP-320-001. If part of the RF compounds will be loaded via a flash evaporation system, this loading must be done prior to the loading via a permeation system.

CAUTION

Do not load the permeation system compounds first; they may elute with the methanol.

3.5.1 Direct the nitrogen flow to the side that will not be used for loading cartridges (see Figure 1) by turning the two stopcocks clockwise, simultaneously.

3.5.2 Loosen the Cajon® fittings and insert the Tenax cartridge with the bottom part down (see Figure 3).

WARNING

Use Kimwipes® when handling the cartridge to avoid contamination.

3.5.3 Tighten the bottom fitting first, then the upper one.

3.5.4 Rotate the two stopcocks clockwise, simultaneously, to direct the gas flow through the cartridge.

3.5.5 Concurrently, start the stopwatch.

3.5.6 Turn the knob on the read-out box to the cartridge flow rate channel. Record this flow rate in the notebook.

3.5.7 Turn the knob to the channel displaying the exhaust flow rate. Record this flow rate in the notebook.

3.5.8 Calculate the time needed to load the amounts desired as follows:

$$t = \left(\frac{G}{P}\right) x \left(\frac{F_1 \qquad F_2}{F_1}\right)$$

(see Section 3.2.1 for definitions).

Perform the calculation based only on the compound whose permeation tube has either the highest or lowest permeation rate.

3.5.9 At the calculated time rotate the two stopcocks to direct gas flow away from the cartridge being loaded.

3.5.10 Handling the cartridge with a Kimwipe®, loosen the Cajon® fittings, the bottom side first.

3.5.11 Remove the cartridge and return it to its culture tube. Seal the tube.

3.5.12 Label the tube. Include the following information: (1) project number; (2) type of mixture loaded; (3) a number indicating the order of loading; and (4) the date. For example:

2190-34-RF-03
5-28-83

3.5.13 Prepare a Chain-of-Custody sheet (COC) sheet with the following information:

CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute, Analytical Sciences Division
Chemistry and Life Sciences Group, Research Triangle Park, NC 27709

SAMPLE CODE 2190-34-RF-03 5-28-83	**TENAX BATCH:**	**VOLUME COLLECTED:**
SAMPLE TYPE:	**TEMPERATURE (°F)**	**VOLUME ANALYZED:**

RELIN-QUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	(Your initials)		Date of Preparation	RF mxt. from permeation system.
(Your initials)				

3.5.14 Analyze by GC/FID the first cartridge loaded, according to the conditions specified in the QA section of SOP-320-001 (Preparation of Tenax).

3.5.15 Refer to the Quality Assurance section in this SOP (Section 3.10) for more details.

3.5.16 To load another cartridge follow instructions in Steps 3.5.2 to 3.5.13.

3.5.17 To change the flow rates to load different amounts of compounds with the same permeation tubes follow instructions in Steps 3.4.14 to 3.4.18.

3.5.18 Allow the system to equilibrate for fifteen minutes before loading.

3.5.19 To load a new cartridge, follow instructions in Steps 3.5.2 to 3.5.13.

3.6 Turning Off the Permeation System

3.6.1 Open the pressure regulator slowly (counter-clockwise) until fully open.

3.6.2 When the back pressure gauge indicates no pressure, open the loading chamber as follows: (1) lower the jack; (2) remove the clamp; (3) carefully move the upper part of the system back; (4) use jack to support the system; and (5) remove the "O" ring.

3.6.3 Turn off the nitrogen flow in the storage chambers.

WARNING

Failure to wear nylon gloves when handling permeation tubes may cause skin damage and/or contamination of the permeation tubes.

3.6.4 With a long glass hook remove one tube at a time, from the loading chamber and transfer immediately to the respective storage chamber.

3.6.5 After the last tube has been transferred reclamp the storage chambers.

3.6.6 With the needle valve that is connected to the "house" nitrogen line, turn the nitrogen gas on.

3.6.7 Reposition the "O" ring on the loading chamber and move the upper part of the system back to the normal position.

3.6.8 Attach the clamp.

3.6.9 Turn off the read-out box.

3.7 Storage of the RF Cartridges

Store the RF cartridges in a sealed paint can in the freezer until they are ready to be loaded with the external standards (see SOP-XXX-YYY).

3.8 Calculations of the Amounts Loaded

3.8.1 For each compound, calculate the amount loaded onto a cartridge using the general formula:

$$G = P \times t \times \frac{F_1}{F_1 + F_2}$$

(see Section 3.2.1 for definitions).

3.8.2 Prepare a table to send to GC/MS/COMP that includes: (1) the number of RF cartridges prepared; (2) the date of preparation; (3) the compounds names; (4) the amounts (in ng/cartridge) loaded for each compound; and (5) the project number (see Table 1 as an example).

3.8.3 Affix a copy of Table 1 in the RTI notebook.

3.9 Loading of External Standards onto RF Cartridges

For the loading of external standards onto RF cartridges refer to SOP-630-003.

3.10 Quality Assurance of the RMR Cartridges

3.10.1 Analyze the first cartridge loaded using a Varian 3700 gas chromatograph equipped with a thermal desorption injection unit and a flame ionization detector and interfaced to a Varian CDS 111 integrator.

3.10.2 Analyze the cartridge using the following instrument conditions:

3.10.2.1 Column: Fused silica capillary column (60 m x 0.25 μm), Liq. phase DB1; or an equivalent column

3.10.2.2 Detector temp: 300°C

3.10.2.3 Injector temp: 280°C

3.10.2.4 Desorption unit temp: 260°C

3.10.2.5 Temp. Program: 30°C (2 min), 4°C/min to 220° (10 min)

3.10.2.6 Attenuation: 128×10^{-12}

3.10.2.7 Recorder chart speed: 1 cm/min

3.10.2.8 He carrier flow: 1.2 mL/min

3.10.2.9 H_2 flow to FID: 30 mL/min

3.10.2.10 Air flow to FID: 300 mL/min

3.10.2.11 Make-up gas: ∿30 mL/min

3.10.3 For each compound, calculate the instrumental response factor using the following formula:

$$\text{Response Factor} = \frac{\text{Area Counts}}{\text{ng loaded}}$$

3.10.4 Compare the calculated response factor with historical response factor generated under identical conditions.

NOTE: The historical data is stored as the mean response factor and standard deviation for each compound. A response factor is considered out-of-control when it deviates from the mean response factor by more than three standard deviations (3σ). This data is stored in a bound RTI notebook, assigned only for the instrument.

3.10.5 If less than two compounds are out-of-control, load the RF cartridges for GC/MS/COMP.

NOTE: Chloroform is not included in this analysis due to its low response on FID.

3.10.6 If two or more compounds are out-of-control, load a second cartridge and analyze it.

3.10.7 If the second one is in control proceed with the loading.

3.10.8 If the second RF cartridge is out-of-control, load and analyze a Column Performance Evaluation (CPM) cartridge. See SOP-630-002 for instructions.

NOTE: The analysis of a CPM cartridge provides a check on the instrument conditions.

3.10.9 If the CPM cartridge is in control, check the permeation system for leaks.

CAUTION

Positive results from a CPM analysis indicates a failure in the permeation system. Be sure the problem is solved before loading the RF cartridges for GC/MS/COMP.

3.10.10 If the CPM cartridge is out-of-control, troubleshoot the instrument before proceeding with the loading.

CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute, Analytical Sciences Division
Chemistry and Life Sciences Group, Research Triangle Park, NC 27709

SAMPLE CODE 2190-34-RF-03 5-28-83	**TENAX BATCH:**	**VOLUME COLLECTED:**
SAMPLE TYPE:	**TEMPERATURE (°F)**	**VOLUME ANALYZED:**

RELINQUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	(Your initials)		Date of Preparation	RF mxt. from permeation system.
(Your initials)				

RTI/ACS-SOP-630-006
May 1984
Revision 0

Standard Operating Procedure for Preparing Response Factor and Column Performance Evaluation Tenax Cartridges Using a Flash Evaporation System

Author:	Approved:	
Nora P. Castillo Nora P. Castillo	CM Sparacino Laboratory Manager, RTI/ACS	6/6/84 Date
	RM Handy QA Officer, RTI/ACS	6/4/84 Date
	E Pellizzari Vice-President, RTI/ACS	6/4/84 Date

Standard Operating Procedure fore Preparing Response Factor and Column Performance Evaluation Tenax Cartridges Using a Flash Evaporation System

RTI/ACS-SOP-630-006

1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is two-fold: 1) to describe the methodology for preparing column performance evaluation (CPMX) and response factor (RF) solutions, and; 2) to describe the loading of Tenax GC cartridges with volatile organic compounds and/or the CPMX components using a flash evaporation system.

This method is applicable to organic compounds whose breakthrough volumes are significantly greater than the diluting solvent. Furthermore, the retention of the solvent by the Tenax must be minimal. Breakthrough volume is defined as that volume of gas required to elute 50% of a compound introduced into one end of a cartridge.

2.0 SUMMARY

The main body of the Flash Evaporation System consists of a glass tube with an injection port. The tube is maintained at a temperature of 250°C with heating tape connected to a variable transformer.

A stream of helium gas transports any solution injected into the port to a sorbent cartridge connected on line (see Figure 1).

3.0 PROCEDURE

3.1 Preparation of a CPMX Solution

A CPMX solution contains compounds from different chemical classes. This solution is used to determine mass calibration and intensity and chromatographic performance of the GC/MS/COMP system.

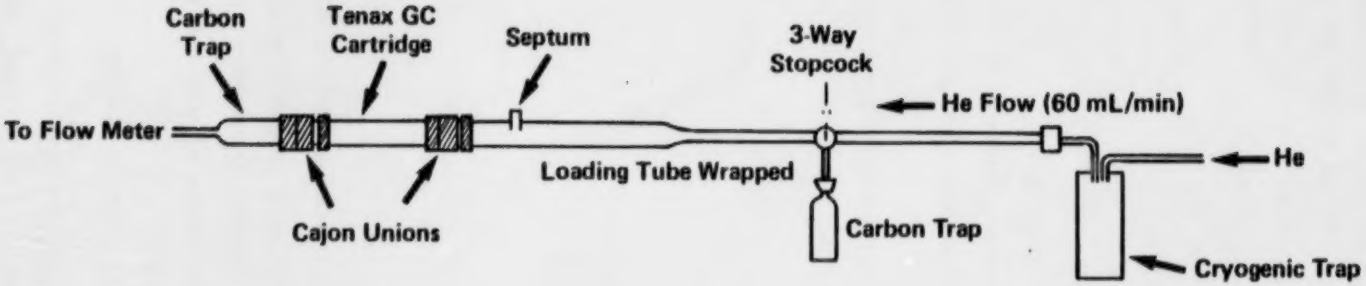
Carbon Trap
Tenax GC Cartridge
Septum
3-Way Stopcock
He Flow (60 mL/min)
To Flow Meter
He
Loading Tube Wrapped
Cajon Unions
Carbon Trap
Cryogenic Trap

Figure 1. Flash evaporation system.

3.1.1 Materials and Instrumentation

3.1.1.1 Capillary pipet, 50 μL or 50 μL syringe
3.1.1.2 Capillary pipet, 100 μL or 100 μL syringe
3.1.1.3 Volumetric flask, 5 mL
3.1.1.4 Volumetric flask, 10 mL
3.1.1.5 Disposable gloves
3.1.1.6 Small spatula
3.1.1.7 Microbalance
3.1.1.8 Pasteur pipets
3.1.1.9 Small aluminum weigh boat
3.1.1.10 Vials with screw cap (open top), 4 mL
3.1.1.11 Silicone rubber septum, Teflon-faced

3.1.2 Reagents

All reagents used are analytical reagent grade (purity >97%).

3.1.2.1 Methyl alcohol, B&J distilled in glass
3.1.2.2 *n*-Octane
3.1.2.3 *n*-Decane
3.1.2.4 5-Nonanone
3.1.2.5 Acetophenone
3.1.2.6 2,6-Dimethylphenol
3.1.2.7 2,6-Dimethylaniline
3.1.2.8 *p*-Xylene
3.1.2.9 *o*-Xylene
3.1.2.10 Ethylbenzene
3.1.2.11 1-Octanol

3.1.3 Procedure

3.1.3.1 In a bound RTI notebook, make a list of the compounds to be used and their respective densities.

3.1.3.2 Calculate the amount required to prepare a stock solution of ~15-20 μg/μL per analyte in a 5 mL volumetric flask.

3.1.3.3 Prepare a table that includes:

Compound Name	Density (mg/μL)	μL Transferred	mg/5 mL	μg/μL

3.1.3.4 Ensure that all the compounds are on hand before preparation of the solution.

WARNING

Use gloves and a hood when handling these chemicals as they may be dangerous to your health.

3.1.3.5 Using a microbalance, weigh the 2,6-dimethylphenol in an aluminum weigh boat and transfer it to the volumetric flask.

WARNING

Do not spend too much time weighing this compound. It is toxic and hygroscopic.

3.1.3.6 Add ~0.5 mL of methyl alcohol to the volumetric flask.

3.1.3.7 Aliquot all liquid compounds in a hood, one at the time, using capillary pipets or syringes.

3.1.3.8 Rinse the walls of the flask with methyl alcohol using a Pasteur pipet to avoid volatility losses.

3.1.3.9 After the last compound has been added, stopper the volumetric flask and invert it five times to mix. Dilute to volume with methyl alcohol.

3.1.3.10 Label the flask as CPMX stock solution. Include the date of preparation and your initials.

3.1.3.11 Mix well. Transfer 100 μL of the stock solution to a 10 mL volumetric flask.

3.1.3.12 Dilute to volume. Mix well and transfer to three screw-cap vials with silicone septa.

3.1.3.13 Label then as CPMX solutions 1, 2, and 3. Include the date of preparation, your initials and the page of the notebook where all the information was recorded.

3.1.3.14 Transfer the stock solution to a screw-cap vial and record all pertinent information.

3.1.3.15 Store all standard solutions at 4°C protected from the light.

3.1.3.16 Open a new vial of the diluted solution every 6 weeks.

3.1.3.17 Use the concentrated stock solution to prepare other dilutions for up to 6 months.

3.2 Preparation of a RF Solution

An RF solution contains all the target compounds of interest for which no permeation tubes are available.

NOTE: Each morning of operation, the Mass Spectrometer operator runs a CPMx and a RF cartridge before beginning the analysis of air or breath samples. This first runs is used to evaluate the conditions of the GC capillary column and to calculate the Relative Response of the Mass Spectrometer to the different compounds.

3.2.1 Materials and Instrumentation

3.2.1.1 Capillary pipet, 50 μL or 50 μL syringe

3.2.1.2 Capillary pipet, 100 μL or 100 μL syringe

3.2.1.3 Volumetric flask, 5 mL

3.2.1.4 Volumetric flask, 10 mL

3.2.1.5 Disposable gloves

3.2.1.6 Small spatula

3.2.1.7 Microbalance

3.2.1.8 Pasteur pipets

3.2.1.9 Small aluminum weigh boat

3.2.1.10 Vials with screw cap (open top), 4 mL

3.2.1.11 Silicone rubber septum, Teflon-faced

3.2.2 Reagents

3.2.2.1 Neat compounds (purity >97%)

3.2.2.2 Methyl alcohol, B&J distilled in glass

3.2.3 Procedure

3.2.3.1 To prepare the stock RF solution, follow the same general instructions as for the CPMX solution (steps 3.1.3.1 to 3.1.3.9 with the exception of step 3.1.3.5).

3.2.3.2 Label the flask as RF stock solution. Include the date of preparation and your initials.

3.2.3.3 Transfer 100 μL of the RF stock solution to a 10 mL volumetric flask.

3.2.3.4 Dilute to volume. Mix well and transfer to three screw-cap vials with silicone septa.

3.2.3.5 Label them as RF solutions. Include the date of preparation, your initials and the page of the notebook where it was recorded.

3.2.3.6 Transfer the stock solution to a screw-cap vial and record all pertinent information.

3.2.3.7 Store the vials at 4°C.

3.2.3.8 Use a new vial every 6 weeks.

3.3 Use of the Flash Evaporation System

3.3.1 Materials and Apparatus

3.3.1.1 Flash evaporation system (see Figure 1)

3.3.1.2 Helium, 2500 psi cylinder (guaranteed 99.995%), with regulator

3.3.1.3 Stopwatch

3.3.1.4 Kimwipes®

3.3.1.5 Hamilton microsyringe, 5 μL

3.3.1.6 Septum, half hole type, low bleed-1

3.3.1.7 Tweezers

3.3.1.8 Insulated gloves

3.3.1.9 Methyl alcohol, B&J distilled in glass

3.3.1.10 Silicone rubber septum, Teflon-faced

3.3.2 Loading of a Tenax GC Cartridge

CAUTION

Be sure the Tenax cartridges are clean before loading them. Follow QA Section of SOP-860-001.

3.3.2.1 Turn on helium gas.

3.3.2.2 After 2 or 3 minutes, fill the cryogenic trap with liquid nitrogen.

WARNING

Introducing the liquid nitrogen before the helium is flowing will cause any water that may be present in the line to freeze and prevent passage of the helium through the system.

3.3.2.3 Rotate the stopcock to direct gas flow through the loading chamber.

3.3.2.4 Adjust the flow with the needle value to approximately 60 mL/min, using the stopwatch and bubble meter.

3.3.2.5 Calculate the flow in mL/min according to the equation:

$$\text{Flow} = \frac{\text{volume (mL)}}{\text{time (sec)}} \times \frac{60 \text{ sec}}{1 \text{ min}}$$

3.3.2.6 Repeat the measurement twice.

3.3.2.7 Rinse the syringe several times with clean methyl alcohol.

3.3.2.8 Put the Tenax cartridge on line with the bottom part toward the loading chamber (see Figure 2).

CAUTION

To avoid contamination, always handle the Tenax cartridge with Kimwipes® or clean nylon gloves.

3.3.2.9 Inject the appropriate volume of the solution to be loaded (normally 2 µL) through the septum, holding the needle in place for 15 seconds.

3.3.2.10 At the same time, start the stopwatch.

3.3.2.11 Pass helium through the cartridge at ~60 mL/min for 12 min (~700 mL total).

NOTE: The breakthrough volume for methyl alcohol is 600 mL.

3.3.2.12 Turn the stopcock to the exhaust position to stop the flow through the system.

3.3.2.13 Return the cartridge to the culture tube.

3.3.2.14 Label the culture tube. Include the project number, solution loaded, a number indicating the order of loading, and the data; for example:

2190-34-RF-02
5/20/83

3.3.2.15 Prepare a Chain-of-Custody sheet (COC sheet) with the following information as shown in Figure 3.

3.3.2.16 To load another cartridge, rotate the stopcock to direct the flow through the system.

3.3.2.17 Follow instructions in steps 3.3.2.7 to 3.3.2.15.

3.3.2.18 To turn off the system, close the helium tank. Maintain the temperature of the system at 250°C.

3.3.2.19 Change the Teflon-silicone septum in the solution vial.

3.3.2.20 Store the solution at 4°C protected from the light.

3.3.2.21 Store the loaded Tenax cartridges in a sealed paint can in the freezer until they are ready to be loaded with the external standards (see SOP-860-001).

3.4 Maintenance of the Flash Evaporation System

3.4.1 Replace the low-bleed septum at the end of the day.

3.4.2 To replace the septum, support the glass tube (250°C) and remove the septum carefully with a pair of tweezers.

3.4.3 Insert a new septum and let it bake overnight before use.

3.4.4 Replace the activated charcoal in the carbon traps every six months.

3.5 Quality Assurance

After a group of cartridges have been loaded, one or two shall be analyzed by GC/FID according to the conditions specified in the QA section of SOP-320-001 (Preparation of Tenax).

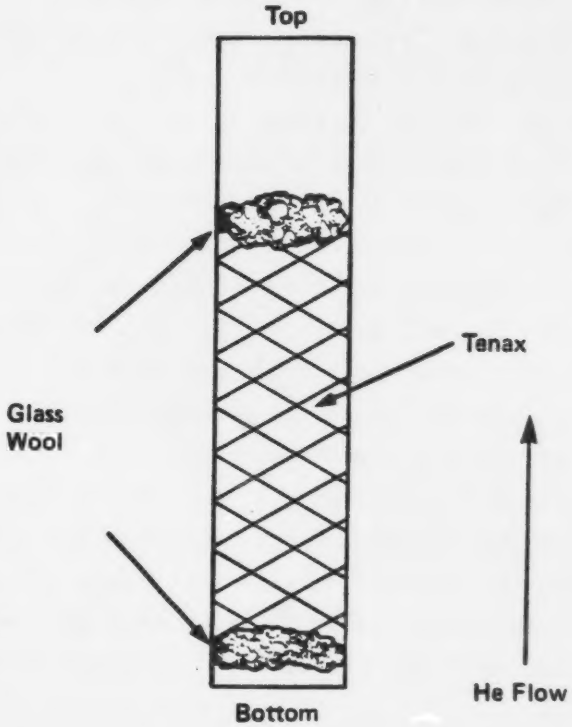
Top
Glass
Wool
Tenax
He Flow
Bottom

Figure 2. Tenax cartridge.

CHAIN-OF-CUSTODY RECORD (TEAM STUDY)

Analytical and Chemical Sciences
Research Triangle Institute, Research Triangle Park, NC 27709

SAMPLE CODE 2190-34-RF-02 5/20/83	**TENAX BATCH:**	**VOLUME COLLECTED:** l
SAMPLE TYPE: TENAX	**TEMPERATURE (°F)**	**VOLUME ANALYZED:**

RELINQUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	your initials		date of preparation	2 μL RF sol. from FES
your initials				

Figure 3. Chain-of-custody sheet.

RTI/ACS-SOP-711-001 (Air)
SOP-717-001 (Breath)
January 1984
Revision 0

Standard Operating Procedure for the Quantitation of Volatile Organic Compounds in Air and Breath Tenax Samples

Author:

Karen A. Brady
Karen A. Brady

Approved:

1/6/84
Laboratory Manager, RTI/ACS Date

1/6/84
QA Officer, RTI/ACS Date

E Pellizzari 1/6/84
Vice-President, RTI/ACS Date

Standard Operating Procedure for the Quantitation of Volatile Organic Compounds in Air and Breath Tenax Samples

RTI/ACS-SOP-711-001 (Air)
SOP-717-001 (Breath)

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure describes the method used to quantitate the target compounds in the TEAM air and breath Tenax samples that have been analyzed by GC/MS. Two BASIC computer programs, QUANT6 and TEAME1, have been written to facilitate the quantitation. The QUANT6 program is used to calculate the concentrations of the targets in each individual sample. TEAME1 is used to edit and store files that are used by QUANT6 to do these calculations. One of these files stores the list of target compounds and, therefore, the list could be modified by editing the file. However, QUANT6 was written specifically for the TEAM matrices, codes and methods of quantitation. To make modifications would require changing or adding to the actual steps of the program. If major modifications are needed, it would be preferable to generate a new program based on QUANT6 rather than alter it.

2.0 SUMMARY OF THE METHOD

The quantitation of air and breath Tenax samples is done using various pieces of information provided by the Mass Spec. Laboratory. After each sample cartridge is analyzed, a data report is prepared listing the ion intensities or nanograms for each target compound identified in the sample. Similar reports are prepared for the blanks and controls from each Tenax batch. Cartridges loaded with known amounts of the targets are analyzed to determine the relative molar response, RMR, of the instrument for each target compound. Finally, limits of detection for the targets for each instrument are determined. All this information is used to determine the concentration of the targets present in the samples.

The RMR values are used to calculate the nanograms of the targets in the blanks, controls and samples. The blanks are used to determine the background present in each batch of Tenax and the controls are used to determine the percent recovery of the targets from the Tenax. The amount of the targets found in each sample are corrected for background and recovery resulting in a value that more accurately represents the amount of the compounds collected onto the Tenax cartridge. These corrected amounts are compared to the limits of detection for the instrument used for the analysis. Any amount that falls below the limit of detection is reported as not detected, ND. Values that lie between the limit of detection and quantifiable limit (defined as 4 times the limit of detection) are reported as trace, T. The values above the quantifiable limit are reported as amounts in the appropriate units of concentration.

3.0 GENERAL ORGANIZATION

3.1 Prepare a Quantitation Log Book

The Quantitation Log Book will be used to record information about the processing and QA of air and breath samples. It will be indicated throughout the SOP what information should be reported, however, it should not be limited to what is recommended here. The purpose of the log book is to have a way to record any information that is relevant to the quantitation process.

3.1.1 Obtain a list of the Tenax batch numbers used and the matrices collected on each batch.

3.1.2 Obtain an RTI Lab Notebook.

3.1.3 In the notebook, designate a section for each Tenax batch used.

3.2 Prepare a TEAM1 Log Book

The TEAME1 Log Book will be used to record changes to the files of the TEAME1 program. Whenever a file is edited, the date, reason for editing and a list of the edited information will be noted in the log book. It is advisable to do the quantitation in an order that minimizes the number of times the files must be edited and, therefore, minimize the possibility of errors in quantitation due to incorrect editing. However, there are times when this is not possible. The purpose of this log book is to keep track of when files are edited and what samples are quantitated using the edited files.

3.2.1 Obtain an RTI Lab Notebook.

4.0 GENERAL INSTRUCTIONS FOR THE USE OF THE COMPUTER

The TEAME1 and QUANT6 programs are stored in the TUCC HP 2000 system. This section gives information on the use of the system. Consult the reference manual if more information is needed.

4.1 Logging On, Running Programs and Logging Off

The specific mechanics of logging on will differ slightly depending on the terminal that is used. The following steps will apply regardless of which terminal is used. (The notes will give some speciifcs on the use of Texas Instruments Silent 700 which could be used, if available.)

4.1.1 Turn the terminal on.

NOTE: The ON/OFF switch is on the back right. Make sure the switch on the right front indicates the terminal is ON LINE.

4.1.2 Dial on the telephone the number 15-549-8751. There will be the normal ringing and then the computer will produce a high-pitched tone.

4.1.3 Place the receiver into the coupler receptacle.

NOTE: A sign at the back of the terminal indicates how the receiver must be positioned in the coupler.

4.1.4 Press the RETURN and LINE FEED keys simultaneously, repeatedly if necessary, and the computer will respond: PLEASE LOG ON. (Failure to get this response indicates the system is down.)

4.1.5 Type HELLO-R100,CLSG,7 and press RETURN. The computer will respond with the system's name, the date and time, and finally: READY.

4.1.6 To use one of the programs, type GET-(the program name) and press RETURN.

4.1.7 Type RUN and press RETURN. The program will begin.

4.1.8 When the work session is finished, log off by typing BYE. The computer will respond with the number of minutes of terminal time used.

4.2 Correcting Errors

In general, if a typing error is made, the mistake must be corrected before the RETURN key is pressed and the information is entered. One exception would be if the information was in an unacceptable format. Then the computer will respond with an error message indicating why it is unacceptable and ask that it be entered again. Individual characters in a line can be deleted by backspacing or the entire line can be deleted.

4.2.1 Deleting individual characters

4.2.1.1 Press the control key, CTRL.

4.2.1.2 Press the letter H once for each backspace needed to make the correction.

4.2.1.3 Release the control key.

NOTE: On the TI Silent 700, the print head will physically move back with each backspace. However, on some terminals it will not and the number of backspaces must be carefully counted.

4.2.2 Deleting entire lines

4.2.2.1 Press the control key, CTRL.

4.2.2.2 Press the letter X.

4.2.2.3 Release the control key.

4.3 Using the Programs

TEAME1 and QUANT6 are interactive programs that request the input of information and responses to yes-or-no questions. When more than one number needs to be entered on one line, the numbers must be separated by a coma. To answer a yes-or-no question, type Y for yes and N for no. After entering any requested information, press RETURN to input the data and advance the program to the next step.

The QUANT6 program can be used by more than one individual at the same time. However, the TEAME1 files cannot be edited while QUANT6 is being used.

5.0 TEAME1

The TEAME1 program is used to edit and store the files accessed by the quantitation program QUANT6. There are four files: 1) Compound Name and Corresponding Ions and MW File, 2) Recovery and Background File, 3) LOD File and 4) RMR File.

5.1 General Instructions

The following are instructions that are common to the editing of all the files. Specific instructions will be given in the following subsections.

5.1.1 Access TEAME1 using the instructions in 4.1.1-4.1.7. The program begins with the following statements:

```
WHICH DATA FILE DO YOU WISH TO EDIT
ENTER 0, 1, 2, 3 or 4
0  = ESCAPE, 1 = COMMAND NAME AND CORRESPONDING IONS
AND MW FILE, 2 = RECOVERY AND BKDG FILE, 3 = LOD FILE AND
4 = RMR FILE.
```

5.1.2 Enter the appropriate number that corresponds to the data file that needs editing. The program will respond with instructions on how to enter the data for that particular file.

NOTE: The choice of 0 = Escape is used, for example, if a mistake is made and the wrong program is accessed.

5.1.3 Enter the data as instructed.

5.1.4 When the editing of the files for Recovery and Background, RMR and LOD is completed, the program will ask if you want a complete listing of the edited data. Always answer yes, Y.

NOTE: This list is used to check that the edited data is correct and complete. A copy will be put in the TEAME1 Log Book.

5.1.5 Check and make sure that the edited data was entered correctly.

5.1.6 The program will ask if you wish to store the data. Type Y or N.

NOTE: If you find that you have made some mistakes in entering the data, you can store the entire data set, access the file again and edit only the incorrect values.

5.1.7 The program will ask if you wish to edit another file. Type Y or N.

5.2 Compound Name and Corresponding Ions and MW File

This file stores the list of the target compounds and their corresponding spectral ions and molecular weights which are used to calculate the amount of each compound in the sample.

5.2.1 Obtain a list of the target compounds for the air and breath samples and the ID number that corresponds to each compound.

5.2.2 Determine the molecular weight of each compound.

5.2.3 Obtain from the Mass Spec. Lab a list of the spectral ion(s) that would be used to quantitate each compound.

5.2.4 Edit the file as described in 5.1.

5.3 Recovery and Background File

This file consists of four separate data sets that store recovery and background information for four Tenax batches.

5.3.1 Calculate the background and recovery for each compound for each Tenax batch as described in 7.4 of this SOP.

5.3.2 Edit the file as described in 5.1.

5.4 LOD File

There are four separate data sets available for the storage of limits of detection. Each LOD set is given a name of no more than ten characters. A code must be established to name the data sets that will describe them and allow them to be distinguished from each other.

5.4.1 Obtain the lists of LODs from the Mass Spec. Lab as instructed in 7.3 of this SOP.

5.4.2 Edit the file as described in 5.1.

5.5 RMR File

This file is also made up of four data sets which store separate RMR sets. Like the LOD data sets, the RMR sets are given names of no more than ten characters. The code chosen to name the RMR sets must describe them and allow them to be distinguished from the other RMR sets.

5.5.1 Calculate the RMRs as described in 7.2 of this SOP.

5.5.2 Edit the file as described in 5.1.

5.6 TEAME1 Log Book

5.6.1 Make photocopies of the lists of edited data.

5.6.2 Record the information in the log book using Appendix, page 1 as an example.

6.0 QUANT6

Using the appropriate files from TEAME1 and the required input from each sample analysis, QUANT6 calculates the concentrations of targets corrected for background and recovery. Then the program, using LOD information, determines whether a compound should be reported as not detected, ND, a trace, T, or an amount. Finally, the program tabulates the pertinent information in a format corresponding to the format of the CCA/Data Entry program.

An example of a run of the program is included in the Appendix (pages 2-4). At the end of each run, the program will ask if you wish to process another sample. If the response is yes, the program will begin again by asking for the sample code. Therefore, the sample will be quantitated using same LOD, RMR and background and recovery data sets as the previous sample. If the response is no, the program will ask if you wish to change any of the parameters and continue. If the response to this question is yes, then the program will start at the beginning. When the response is negative, the computer will print DONE and you may either log off or access another program.

6.1 General Information

The program will ask for the analyst and transcriber id numbers. These numbers are the individuals' RTI identification numbers. The analyst ID number will be written on each Mass Spec. data report form. The sample code must be entered without any spaces or dashes. The program will ask for a start time and finish time which are the sample collection times. These times can be found on the SP/COC sheets and will be included as part of the Mass Spec. data report. Breath samples have only one time recorded and it will be reported on the data summary sheet as the finish time. (When asked to enter the start time, type a dash, -.)

6.2 Calculating Nanograms Per Cartridge

Part of the program output lists uncorrected nanograms/cartridge found for each compound. Therefore, the program can be used to calculate the nanograms found per cartridge for blanks, controls and audit samples. However, this means that there will be input requested that is irrelevant to a calculation of nanograms using ion intensity.

When asked to select an LOD or background and recovery data set, any one can be chosen since they are not used in the calculation. Collection times and dates, sampling volume and temperature do not apply to blanks controls and audit samples and, therefore, any number can be entered.

> NOTE: The reason for entering false information is simply to make the program run. Look carefully at the example run in the Appendix and note where the required input is to be entered. Make sure you understand what is needed and what is irrelevant.

In 6.1 the instruction was given to enter the sample code without spaces or dashes. This is because the matrix code must appear in spaces 7 and 8. Because the blanks and controls do not have the same codes as the samples, it may be necessary to enter a code with spaces or dashes in order that matrix code be entered in spaces 7 and 8.

6.3 Correcting Errors

After the samples are quantitated, a certain percentage must be checked for correctness. This involves checking each step of the run.

Therefore, it is important that all the entries be clear and legible. Typing errors can be corrected by either backspacing or deleting a line as described in 4.2. Any information that appears on the data summary sheet (e.g., sample code, dates, collection times, id numbers) can be corrected by backspacing. Although this may make the entry illegible, it is still possible to verify that the information is correct. In general, it is preferable to delete an incorrect line rather than backspacing and leaving the entry unclear.

When entering the ion intensities or nanograms for each compound, each time the target ID number is entered any information previously entered is deleted. This is the only opportunity in the program to correct a mistake after the RETURN key has been pressed.

7.0 PREPARATION

7.1 Receive and Organize Mass Spec. Data Report Forms

After each sample, blank or control cartridge is analyzed by GC/MS, a data report form is prepared. The data will be reported as either ion intensities per mass spectral ion per compound or nanograms per mass spectral ion per compound (See Appendix, pages 5-6). Along with the sample, blank and control cartridges that are prepared for and analyzed by GC/MS, there are a number of cartridges with sample codes for duplicate field samples which are EPA audit samples. As data report forms are received from the Mass Spec. Lab, they should be separated and organized in the following manner.

7.1.1 EPA Audit Samples

7.1.1.1 Obtain from the QA officer a list of air and breath sample codes that were used to disguise EPA audit samples.

7.1.1.2 Look through the Mass Spec. data report forms for duplicates and identify any EPA audit samples.

7.1.1.3 Separate these reports from the actual sample reports and check the samples off on the list.

7.1.1.3.1 If data is reported in nanograms per compound, give these reports to the QA officer.

7.1.1.3.2 If the data is reported in ion intensities, calculate the nanogram amounts using the QUANT6 program and give the results to the QA officer. (See 6.2 of this SOP.)

7.1.2 Blanks and Controls

7.1.2.1 Obtain a list of all the blanks and controls in each Tenax batch that were exposed in the field and analyzed by GC/MS.

7.1.2.2 Separate the blanks and controls from the sample data report forms and check them off the list.

7.1.2.3 Put blanks and controls in groups by Tenax batch.

7.1.3 Field Samples

7.1.3.1 Divide sample report forms into groups such that the samples in each group have all of the following in common: 1) city (if applicable), 2) sample matrix, 3) mass spec. instrument used for analysis and 4) Tenax batch.

7.1.3.2 In the appropriate sections of the Quantitation Log Book, note which samples have been received.

7.2 Calculate Mean RMR Values

This section applies only for quantitation done using ion intensities.

The RMR (Relative Molar Response) for any compound, x, and standard, s, is defined as:

$$RMR_x = \frac{I_x/\text{moles}_x}{I_s/\text{moles}_s}$$

where I = the intensity of a characteristic ion.

Each day that samples are analyzed, cartridges loaded with known amounts of the target compounds and the standard are run and RMR values are calculated based on the results. These values are put into groups that are established based on any changes over time in the performance of the instrument. A mean RMR value is calculated for each compound for each time period. The mean RMR values are used to quantitate the samples, blanks and controls that were run during that time period.

7.2.1 Obtain a list of the RMR values from each instrument used for sample analysis.

7.2.2 Obtain copies of the instrument log books covering the period of time which samples were run.

7.2.3 For each instrument, use the log book to determine how the RMR groups should be divided. A new RMR group shall be established each time a physical change was made to the instrument or a change in response was noted.

7.2.4 For each time period, calculate a mean RMR for each mass spectral ion for each compound using the form in the Appendix, page 7.

NOTE: SDU stands for standard deviation unit and is defined as 10% of the RMR mean.

7.2.4.1 If more than 50% of the RMR values fall outside the range, then a corrected RMR mean should not be calculated and the preliminary RMR mean will be used for quantitation.

7.3 Limits of Detection

The limit of detection for a compound is the minimum amount that can be identified under specific instrument conditions (i.e. temperature program, column, etc.).

7.3.1 Obtain a list of the limits of detection from each instrument used for sample analysis.

7.4 Calculate Mean Backgrounds and Mean Recoveries for Each Tenax Batch

The backgrounds and recoveries for a particular Tenax batch are determined using the field blanks and control sets exposed in the field. A control set is composed of two cartridges loaded with the target compounds, one from the permeation system (PS) and one from the flash evaporation system (FES).

When the data for blanks and controls is reported as ion intensities, the program, QUANT6, is used to calculate the nanogram amounts of the compounds found in each cartridge.

7.4.1 Calculate the nanogram amounts of the targets in each blank and control

7.4.1.1 Based on the dates they were analyzed and on the instrument used, determine which RMR groups will be needed to do the calculation.

7.4.1.2 Use TEAME1 to enter and store the needed RMR sets.

7.4.1.3 Calculate the nanograms of each compound per cartridge using QUANT6 as described in 6.2 of this SOP.

7.4.2 Calculate Mean Background

7.4.2.1 Make sure that there is data available for each blank sent to the Mass Spec. Lab using the list obtained in 7.1.2.1.

7.4.2.2 Fill out a Background table entering the nanogram amounts found for each target in each blank. (See Appendix, page 8.)

7.4.2.3 For each compound, calculate and record the mean background, standard deviation and coefficient of variation.

7.4.3 Calculate Mean Recovery

7.4.3.1 Obtain a list of the amounts of the target compounds loaded in each type of control.

7.4.3.2 Make sure that there is data available for each control sent to the Mass Spec. Lab using the list obtained in 7.1.2.1.

7.4.3.3 Fill out a Recovery table entering the nanograms found for each target loaded in each control (See Appendix, page 9).

NOTE: The nanograms found for the compounds not loaded may be included in the table written in pencil. These values will not be used to calculate recovery but could be used to trace a problem with contamination, if one should occur.

7.4.3.4 After all the controls have been tabulated, list in the next column the mean background calculated for each compound.

7.4.3.5 In the next column, list the amount loaded for each compound.

NOTE: If the mean background is greater than the amount loaded, a recovery will not be calculated and the compound will be reported as NC on the data summary sheet.

7.4.3.6 For each compound, calculate and record the mean recovery, standard deviation and coefficient of variation.

NOTE: Mean recovery is defined as:

$$\frac{\text{Mean of amounts found in controls - Mean background}}{\text{Amount loaded}}$$

8.0 SAMPLE QUANTITATION

Sample quantitation can begin with any of the groups of data forms established in 7.1.3.1 for which all the needed information is available.

NOTE: If possible, establish a plan for sample quantitation that will minimize the number of times the TEAME1 files must be edited. However, the ability to do this is limited by the availability of all the various pieces of information that are needed.

8.1 Based on the dates of analysis, divide the sheets into RMR groups.

8.2 Using TEAME1, enter and store the needed data files. Make the appropriate entries into the TEAME1 Log Book.

8.3 Quantitate the samples as described in 6.0. Make the appropriate entries in the Quantitation Log Book.

9.0 QUALITY ASSURANCE CHECK

Twenty-five percent of the runs must be checked for correctness by another individual.

9.1 Instructions for QA Check

9.1.1 Obtain from the transcriber a group of Mass Spec. data report forms and their corresponding QUANT6 runs. You will also receive or request any other information needed to complete the QA process, such as a list of the time periods covered by each RMR group.

9.1.2 Randomly select 25% of the samples and check the QUANT6 run carefully against the data report form. Initial each QUANT6 run you check. (Do not put initials on the data summary sheet.)

9.1.2.1 Check the first sample run and each subsequent sample where the program is restarted to make certain that the three TEAME1 data files selected are correct.

9.1.3 Make a list of any errors found.

9.1.4 Return the QUANT6 runs and data report forms to the transcriber along with the list of errors if any were found.

9.1.5 Make an entry in the appropriate section of the Quantitation Log Book indicating what samples were QA checked, if any errors were found and what those errors were. Initial the log book entry.

9.2 Make Corrections

9.2.1 Any errors found in the QA check will be corrected.

9.2.2 Note in the Quantitation Log Book that the errors were corrected.

10.0 PREPARATION FOR BATCHING

The data summary sheets must be submitted to CCA in groups called batches.

10.1 Obtain a copy of the sampling schedule for the sampling trip.

10.2 Make photocopies of the data summary sheet portion of the print-out.

10.3 Put the data summary sheets into groups by matrix and in numerical order.

10.4 Check off on the schedule each sample for which there is a data summary sheet.

10.5 Submit the data summary sheets to the individual responsible for batching.

10.6 After all the data summary sheets for a particular matrix have been submitted for batching, check the sampling schedule and determine if any data is missing.

10.6.1 If there is data missing, determine whether data is available in Mass Spec. or whether the sample was lost or broken.

NOTE: If a sample was scheduled to be collected and was either not collected, lost or broken, all the compounds for that sample will be reported NC.

APPENDIX

TEAME1 LOG BOOK

Date: 3/25/83
File: Background and Recovery
Data Set #: 3
Samples: New Jersey II XV

```
ID#  NAME                                        BACKGROUND  RECOVERY
 1    VINYLIDENE CHLORIDE                         4           .52
 2    CHLOROFORM                                  102         .36
 3    1,2-DICHLOROETHANE                          3           .95
 4    1,1,1-TRICHLOROETHANE                       63          .76
 5    BENZENE                                     173         .78
 6    CARBONTETRACHLORIDE                         4           .8
 7    TRICHLOROETHYLENE                           117         .78
 8    BROMODICHLOROMETHANE                        0           1.09
 9    DIBROMOCHLOROMETHANE                        0           .88
 10   TOLUENE                                     0           1
 11   TETRACHLOROETHYLENE                         31          .94
 12   CHLOROBENZENE                               8           1.03
 13   BROMOFORM                                   0           .87
 14   DIBROMOCHLOROPROPANE                        0           1.04
 15   STYRENE                                     6           .97
 16   M-DICHLOROBENZENE                           0           1
 17   P-DICHLOROBENZENE                           2           1.16
 18   O-DICHLOROBENZENE                           2           1.13
 19   ETHYLBENZENE                                20          .97
 20   O-XYLENE                                    20          .86
 21   M-XYLENE                                    0           1
 22   P-XYLENE                                    58          .84
WHICH TENAX BATCH DOES THIS DATA CORRESPOND TO?
?49
```

Date: 4/10/83
File: RMR
Data Set #2
Samples: New Jersery II AV

```
NJ2 FIN P1
ID# NAME
                                    IONS                  RMP VALUES
 VINYLIDENE CHLORIDE           61  96  98         0.340 0.120 0.077
 CHLOROFORM                    83  85   0         0.680 0.420 0.000
 1,2-DICHLOROETHANE            62  64   0         0.630 0.200 0.000
 1,1,1-TRICHLOROETHANE         61  97  99         0.340 0.590 0.340
 BENZENE                       78   0   0         0.620 0.000 0.000
 CARBONTETRACHLORIDE          117 119   0         0.550 0.490 0.000
 TRICHLOROETHYLENE             95 130 132         0.410 0.390 0.390
 BROMODICHLOROMETHANE          83 129   0         0.610 0.043 0.000
 DIBROMOCHLOROMETHANE         127 129   0         0.430 0.580 0.000
 TOLUENE                        0   0   0         0.000 0.000 0.000
 TETRACHLOROETHYLENE          164 166   0         0.420 0.550 0.000
 CHLOROBENZENE                112 114   0         0.610 0.190 0.000
 BROMOFORM                    173   0   0         0.610 0.000 0.000
 DIBROMOCHLOROPROPANE         155 157   0         0.360 0.470 0.000
 STYRENE                      104   0   0         0.590 0.000 0.000
 M-DICHLOROBENZENE              0   0   0         0.000 0.000 0.000
 P-DICHLOROBENZENE            146 148   0         0.950 0.630 0.000
 O-DICHLOROBENZENE            146 148   0         0.870 0.590 0.000
 ETHYLBENZENE                  91 106   0         1.140 0.290 0.000
 O-XYLENE                      91 106   0         1.010 0.370 0.000
 M-XYLENE                       0   0   0         0.000 0.000 0.000
 P-XYLENE                      91 106   0         1.090 0.380 0.000
YOU WANT TO STORE THIS RMP DATA SET ?
```

Reproduced from
best available copy.

```
LEASE LOG IN
HELLO-R100,CLSC,7


TUCC HP 2000/ACCESS SYSTEM #1

05-20-83 09:40 AM  PORT #03

READY

GET-QUANT6
RUN
QUANT6

THE CURRENT LOD DATA SETS ARE:
DATA SET # 1     CONTAINS THE LOD DATA FOR NJ3 F3300
DATA SET # 2     CONTAINS THE LOD DATA FOR NJZLKS F4*
DATA SET # 3     CONTAINS THE LOD DATA FOR NJZFIN F5*
DATA SET # 4     CONTAINS THE LOD DATA FOR NJZFIN F6*
WHICH LOD DATA SET DO YOU WISH TO ACCESS?
?1
THE CURRENT BKGD AND RECOVERY DATA SETS HOLD DATA
FOR THE SPECIFIED TENAX BATCHES.
DATA SET # 1     CONTAINS THE DATA FOR BATCH 6
DATA SET # 2     CONTAINS THE DATA FOR BATCH 43
DATA SET # 3     CONTAINS THE DATA FOR BATCH 46
DATA SET # 4     CONTAINS THE DATA FOR BATCH 47
ENTER THE DATA SET # YOU WISH TO ACCESS
?1
DO YOU WANT A LISTING OF THE BACKGROUND AND RECOVERY BY COMPOUND
?Y
ID#  NAME                                BACKGROUND  RECOVERY
 1    VINYLIDENE CHLORIDE                 1           .7
 2    CHLOROFORM                          9           1.08
 3    1,2-DICHLOROETHANE                  2           1.07
 4    1,1,1-TRICHLOROETHANE               36          1
 5    BENZENE                             10          .75
 6    CARBONTETRACHLORIDE                 0           .96
 7    TRICHLOROETHYLENE                   11          1.21
 8    BROMODICHLOROMETHANE                1           1.47
 9    DIBROMOCHLOROMETHANE                0           1.27

 10   TOLUENE                             0           1
 11   TETRACHLOROETHYLENE                 1           .69
 12   CHLOROBENZENE                       0           .9
 13   BROMOFORM                           0           .99
 14   DIBROMOCHLOROPROPANE                0           .75
 15   STYRENE                             0           .34
 16   M-DICHLOROBENZENE                   0           1
 17   P-DICHLOROBENZENE                   0           .64
 18   O-DICHLOROBENZENE                   0           .60
 19   ETHYLBENZENE                        1           .63
 20   O-XYLENE                            1           .53
 21   M-XYLENE                            0           1
 22   P-XYLENE                            3           .67
DO YOU WISH TO PROCESS A SAMPLE?
?Y
DO YOU WANT TO CALCULATE USING NG FOUND OR ION INTENSITIES?
ENTER 1=NG FOUND, 2=ION INTENSITIES
?1
ENTER THE TRANSCRIPTION DATE,IE TODAY'S DATE
?5/20/83
ENTER IN THE TRANSCIBER ID
?6326
BAD INPUT, RETYPE FROM ITEM 1
??6326
ENTER THE SAMPLE CODE
?310110AVF1
ENTER THE DATE THE SAMPLE WAS COLLECTED
?1/28/83
ENTER THE DATE THE SAMPLE WAS ANALYZED
?2/14/823
ENTER THE ANALYST ID#
?2702
ENTER THE START TIME
?19:6
ENTER THE FINISH TIME
?0707
enterin the compound id#, then enter in the ng
of the compound found in the sample after the computer
```

```
enterin the compound id#, then enter in the ng
of the compound found in the sample after the computer
responds with the name of the compounc.  enter 0 for the
id# when the data entry is complete
id#
?1
VINYLIDENE CHLORIDE  NG FOUND =?6
id#
?9
DIBROMOCHLOROMETHANE  NG FOUND =?2
id#
?15
STYRENE  NG FOUND =?88
id#
?17
P-DICHLOROBENZENE  NG FOUND =?598
id#
?18
O-DICHLOROBENZENE  NG FOUND =?5
id#
?19
ETHYLBENZENE  NG FOUND =?228
id#
?20
O-XYLENE  NG FOUND =?261
id#
?22
P-XYLENE  NG FOUND =?704
id#
?2
CHLOROFORM  NG FOUND =?114
id#
?3
1,2-DICHLOROETHANE  NG FOUND =?17
id#
?4
1,1,1-TRICHLOROETHANE  NG FOUND =?719
id#
?5
BENZENE  NG FOUND =?504
id#
?6
CARBONTETRACHLORIDE  NG FOUND =?25
id#
?7
TRICHLOROETHYLENE  NG FOUND =?196
id#
?8
BROMODICHLOROMETHANE   NG FOUND =?81
id#
?11
TETRACHLOROETHYLENE  NG FOUND =?485
id#
?12
CHLOROBENZENE  NG FOUND =?16
id#
?12
CHLOROBENZENE  NG FOUND =?14
id#
?56
id#
?0
ENTER THE TEMPERATURE AND VOLUME RESPECTIVELY
?67,21.6
do you want a list of the corrected quantity of compounds found??
ID  NAME                              NG       BKGD       RCVY      [illegible]
                                   FOUND       SUBT       CORR
 1 VINYLIDENE CHLORIDE                 6          5          7      [illegible]
 2 CHLOROFORM                        114        105        100      [illegible]
 3 1,2-DICHLOROETHANE                 17         15         12      [illegible]
 4 1,1,1-TRICHLOROETHANE             719        687        66[illegible]  [illegible]
 5 BENZENE                           504        474 [illegible]     [illegible]
 6 CARBONTETRACHLORIDE                25         25         2[illegible]  [illegible]
 7 TRICHLOROETHYLENE                 196        185        147      21.6
 8 BROMODICHLOROMETHANE               81         60         56      21.6
 9 DIBROMOCHLOROMETHANE                2          2          2      [illegible]
10 TOLUENE                             0          0          0      21.6
11 TETRACHLOROETHYLENE               485        464        544      21.6
12 CHLOROBENZENE                      14         14         16      21.6
```

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```
  6 CARBONTETRACHLORIDE           25        25        26      21.8
  7 TRICHLOROETHYLENE            196       185       147      21.8
  8 BROMODICHLOROMETHANE          81        80        56      21.8
  9 DIBROMOCHLOROMETHANE           2         2         2      21.8
 10 TOLUENE                        0         0         0      21.8
 11 TETRACHLOROETHYLENE          485       464       544      21.8
 12 CHLOROBENZENE                 14        14        16      21.8
 13 BROMOFORM                      0         0         0      21.8
 14 DIBROMOCHLOROPROPANE           0         0         0      21.8
 15 STYRENE                       88        88       105      21.8
 16 M-DICHLOROBENZENE              0         0         0      21.8
 17 P-DICHLOROBENZENE            598       596       712      21.8
 18 O-DICHLOROBENZENE              5         5         6      21.8
 19 ETHYLBENZENE                 228       227       259      21.8
 20 O-XYLENE                     261       260       295      21.8
 21 M-XYLENE                       0         0         0      21.8
 22 P-XYLENE                     704       701       806      21.8
```

```
                     2190 TEAM DATA SUMMARY SHEET
                           FOR VOLATILES

SAMPLE CODE: 310110 AV F1                    MATRIX: AIR
-----------------------------------------------------------------------
       COMPOUND                        AMOUNT          LOD          QL
-----------------------------------------------------------------------
  1 VINYLIDENE CHLORIDE               T                          13 E-02

  2 CHLOROFORM                         46 E-01

  3 1,2-DICHLOROETHANE                T                          55 E-02

  4 1,1,1-TRICHLOROETHANE              44 E+00

  5 BENZENE                            29 E+00

  6 CARBONTETRACHLORIDE                12 E-01

  7 TRICHLOROETHYLENE                  67 E-01

  8 BROMODICHLOROMETHANE               26 E-01

  9 DIBROMOCHLOROMETHANE              ND            18 E-02

 10 TOLUENE                           NC

 11 TETRACHLOROETHYLENE                25 E+00

 12 CHLOROBENZENE                      71 E-02

 13 BROMOFORM                         ND            23 E-02

 14 DIBROMOCHLOROPROPANE              ND            28 E-02

 15 STYRENE                            48 E-01

 16 M-DICHLOROBENZENE                 NC

 17 P-DICHLOROBENZENE                  33 E+00

 18 O-DICHLOROBENZENE                 T                          37 E-02

 19 ETHYLBENZENE                       12 E+00

 20 O-XYLENE                           14 E+00

 21 M-XYLENE                          NC

 22 P-XYLENE                           37 E+00

-----------------------------------------------------------------------
ANALYST ID:  2702  TRANSCRIBER ID:  6328
DATE COLLECTED:     1/28/83   VOLUME ANALYZED:  21.8 LITER
DATE ANALYZED:      2/16/83    TEMP:   67 F
DATE TRANSCRIBED:   5/20/83   COLLECTION TIMES-START: [illegible] FINISH: [illegible]
```

Project - 2190 (TEAM)
Elizabeth and Bayonne, NJ/Greensboro, NC
AIR AND BREATH

Tenax Batch 45
GC/MS Model No. 3300

TEAM 21401-5 AV-F2 File# 3AABF054

PFB (ng) 128 **Sampling Temp. (°F)** 80 **Date Collected** 7-9-82
PFT (ng) 133 **Sampling Volume** 20.6 **Date Analyzed** 8-30-82
Collection Times: Start 7:05 **Finish** 16:59 **Analyst ID** 2702

MP Code No.	TSO Code No.	Compound	M.W.	Scan No.	Ions	Areas	Ratios (2°/1°; 3°/1°)			ng	μg/m³
							Th.	Obs.	Clean		
	18	Perfluorobenzene	186	291	186 117	19231 20571					
		Perfluorotoluene	236	337	69 79 93 117 148 167 186 217 236	— 2107 3132 9239 1230 2399 11074 19162 9557			69 No		
1	117	Vinylidene chloride	96	NF e143	*61 96 98	Trace	.62 .38				
2	125	Chloroform	118	301	*83 85	12746 7855	.62				
3	133	1,2-Dichloro-ethane	98	335	*62 64	1146 355	.32				
4	141	1,1,1-Trichloro-ethane	132	347	61 *97 99	24152 44912 24859	.58 .62				
5	158	Benzene	78	370		237811					
6	166	Carbon tetra-chloride	152	377	*117 119	1612 1347	.95				
7	174	Trichloro-ethylene	130	426	*95 130 132	38323 35450 32459	1.00 .95				
8	182	Bromodichloro-methane	162	NF	*83 129		.18				
9	190	Dibromochloro-methane	206	NF	127 *129		.78				
10	208	Toluene	92	533	91	767499					
11	216	Tetrachloro-ethylene	164	603	164 *166	25990 32469	.78				
12	224	Chlorobenzene	112	648	*112 114	5337 1103	.32				
13	232	Bromoform	250	NF	173						
14	240	Dibromochloro-propane	234	NF	155 *157		.78				
15	257	Styrene	104	714	104	5065					
17	273	*m* & *p*-Dichloro-benzene	146	878	*146 148	6952 4058	.65				
18	281	*o*-Dichloro-benzene	146	907	*146 148	702 319	.65				
19	299	Ethylbenzene	106	675	*91 106	82412 22416	.32				
20	307	*o*-Xylene	106	721	*91 106	70216 28611	.58				
22	323	*m* & *p*-Xylene	106	688	*91 106	203149 86963	.66				
		d_6-Benzene	84	NF	84						
		d_5-Bromo-ethane	113	NF	113 115						

PROJECT NO. 2190

SUBJECT TEAM 31011-0 AV-F1 B#60 DATE 2-16-83

File# 3AAAF852

1600 EM

TEAM 31011-0

AV-F1

PFB = 128

PFT < 133

Temp 67°F

Vol Col 21.8

Date Col 1-27-83

19:16 - 7:03

```
QUANTITATION REPORT    FILE: 3AAAF852

DATA: 3AAAF852.TI
02/16/83 21:35:00
SAMPLE: TEAM 31011-0 AV-F1,(B#60),1600EM
SUBMITTED BY: EP              ANALYST: PBD      2702

AMOUNT=AREA(HGHT) * REF.AMNT/(REF.AREA(HGHT)* RESP.FACT)
RESP. FAC. FROM  LIBRARY ENTRY
                                                                  Code*
NO  NAME
 1  PERFLUOROBENZENE         NO  M/E  SCAN   TIME  REF    RRT  METH      AREA(HGHT)  AMOUNT           %TOT
 2  PERFLUOROBENZENE          1  117   289   9:38    2  1.000  A BV        22354.    128.584 NG/CT   3.26
 3  VINYLIDENE CHLORIDE       2  186   289   9:38    2  1.000  A BV        25144.    128.000 NG/CT   3.25
 4  VINYLIDENE CHLORIDE       3   61   188   6:16    2  0.651  A VV   1      787.  6   5.686 NG/CT   0.14
 5  VINYLIDENE CHLORIDE       4   96   187   6:14    2  0.647  A BB          670.      7.344 NG/CT   0.19
 6  DIBROMOCHLOROMETHANE      5   98   188   6:16    2  0.651  A BB          181.      2.878 NG/CT   0.07
 7  DIBROMOCHLOROMETHANE      6  127   556  18:32    2  1.924  A BB          139.      1.382 NG/CT   0.04
 8  STYRENE                   7  129   556  18:32    2  1.924  A BB   9      225.  2   1.707 NG/CT   0.04
 9 m P-DICHLOROBENZENE        8  104   712  23:44    2  2.464  A BV   15   42035. 88 87.699 NG/CT   2.22
10  P-DICHLOROBENZENE         9 m146   865  28:50    2  2.993  A BB         1441.      2.958 NG/CT   0.08
11 m P-DICHLOROBENZENE       10 p146   872  29:04    2  3.017  A BV   17  289684. 595 594.632 NG/CT  15.08
12  P-DICHLOROBENZENE        11 m148   865  28:50    2  2.993  A BB          915.      2.823 NG/CT   0.07
13  O-DICHLOROBENZENE        12 p148   872  29:04    2  3.017  A BV       186816.    573.900 NG/CT  14.56
14  O-DICHLOROBENZENE        13  146   901  30:02    2  3.118  A BB   18    1046.  5   5.279 NG/CT   0.13
15  ETHYLBENZENE             14  148   901  30:02    2  3.118  A BB         1004.      4.331 NG/CT   0.11
16  ETHYLBENZENE             15   91   674  22:28    2  2.332  A BB   19  186917. 228 228.186 NG/CT   5.79
17  O-XYLENE                 16  106   674  22:28    2  2.332  A BB        54393.    229.841 NG/CT   5.83
18  O-XYLENE                 17   91   719  23:58    2  2.488  A BV   20  186555. 261 260.904 NG/CT   6.62
19  P-XYLENE                 18  106   719  23:58    2  2.488  A BV        79915.    257.481 NG/CT   6.53
20  P-XYLENE                 19   91   687  22:54    2  2.377  A BB   2   617290. 704 704.579 NG/CT  17.87
                             20  106   687  22:54    2  2.377  A BV       276920.    715.611 NG/CT  18.15
QUANTITATION REPORT    FILE: 3AAAF852

DATA: 3AAAF852.TI
02/16/83 21:35:00
SAMPLE: TEAM 31011-0 AV-F1,(B#60),1600EM
SUBMITTED BY: EP              ANALYST: PBD

AMOUNT=AREA(HGHT) * REF.AMNT/(REF.AREA(HGHT)* RESP.FACT)
RESP. FAC. FROM  LIBRARY ENTRY

NO  NAME
 1  PERFLUOROBENZENE (INT STD)   NO  M/E  SCAN   TIME  REF    RRT  METH      AREA(HGHT)  AMOUNT           %TOT
 2  PERFLUOROBENZENE (INT STD)    1  117   289   9:38    2  1.000  A BV        22354.    125.465 NG/CT   2.58
 3  CHLOROFORM                    2  186   289   9:38    2  1.000  A BV        25144.    128.000 NG/CT   2.63
 4  CHLOROFORM                    3   83   299   9:58    2  1.035  A BV   2    34025. 114 113.970 NG/CT   2.34
 5  1.2-DICHLOROETHANE            4   85   299   9:58    2  1.035  A BV        22976.    117.314 NG/CT   2.41
 6  1.2-DICHLOROETHANE            5   62   334  11:08    2  1.156  A BV   3     2074.  17  17.219 NG/CT   0.35
 7  1.1.1-TRICHLOROETHANE         6   64   334  11:08    2  1.156  A BB         1056.     17.692 NG/CT   0.36
 8  1.1.1-TRICHLOROETHANE         7   61   345  11:30    2  1.194  A BV        66205.    654.421 NG/CT  13.43
 9  1.1.1-TRICHLOROETHANE         8   97   345  11:30    2  1.194  A BV   4   163329. 717 719.340 NG/CT  14.76
10  BENZENE                       9   99   345  11:30    2  1.194  A BV        97963.    726.963 NG/CT  14.92
11  CARBON TETRACHLORIDE         10   78   368  12:16    2  1.273  A BB   5   407000. 504 503.078 NG/CT  10.34
12  CARBON TETRACHLORIDE         11  117   375  12:30    2  1.298  A BV   6     4152.  25  25.043 NG/CT   0.51
13  TRICHLOROETHYLENE            12  119   375  12:30    2  1.298  A BB         4009.     27.246 NG/CT   0.56
14  TRICHLOROETHYLENE            13   95   424  14:08    2  1.467  A BB   7    32356. 196 196.091 NG/CT   4.02
15  TRICHLOROETHYLENE            14  130   424  14:08    2  1.467  A BB        31272.    199.994 NG/CT   4.10
16  BROMODICHLOROMETHANE         15  132   424  14:08    2  1.467  A BB        31556.    202.065 NG/CT   4.15
17  BROMODICHLOROMETHANE         16   83   423  14:06    2  1.464  A VB   8    12266.  81  80.882 NG/CT   1.66
18  TETRACHLOROETHYLENE          17  129   422  14:04    2  1.460  A BB          296.     25.540 NG/CT   0.52
19  TETRACHLOROETHYLENE          18  164   602  20:04    2  2.083  A BB        40991.    487.104 NG/CT  10.00
20  CHLOROBENZENE                19  166   602  20:04    2  2.083  A BB   11   64117. 495 484.991 NG/CT   9.95
21  CHLOROBENZENE                20  112   649  21:38    2  2.246  A BB   12    2003.  14  13.989 NG/CT   0.29
                                 21  114   647  21:34    2  2.239  A BB          294.      4.891 NG/CT   0.10
```

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DETERMINATION OF RMR VALUES FOR SAMPLE QUANTITATION

Instrument: ______________________________

Time Period: ______________________________

Compound: ______________________________

Mass: ____________________

Prel. RMR Mean (N =   ): ____________________

SDU (Assume 10% S.D.): ____________________

3.0 (S.D.U.): ______________

RMR Mean ± 3.0 (S.D.U.): __________ TO __________

RMR Values Outside Range:

Date	Value	Date	Value

Corr. RMR Mean: ______________________________

S.D.: ____________________

C.V.: ____________________

Background

Tenax Batch

Code Number																
Date Run																
Instrument																
1 vinylidene chloride																
2. chloroform																
3. 1,2-dichloroethane																
4. 1,1,1-trichloroethane																
5. benzene																
6 carbon tetrachloride																
7 trichloroethylene																
8 bromodichloromethane																
9 dibromochloromethane																
10. toluene																
11 tetrachloroethylene																
12 chlorobenzene																
13. bromoform																
14 dibromochloropropane																
15. styrene																
16 m-dichlorobenzene																
17 p-dichlorobenzene																
18 o-dichlorobenzene																
19 ethylbenzene																
20 o-xylene																
21 m-xylene																
22 p-xylene																
Intensity of PFB (184)																
Amount PFB loaded																

Recovery

Tenax Batch:

Code Number																
Date Run																
Instrument																
1 vinylidene chloride																
2 chloroform																
3 1,2-dichloroethane																
4 1,1,1-trichloroethane																
5 benzene																
6 carbon tetrachloride																
7 trichloroethylene																
8 bromodichloromethane																
9 dibromochloromethane																
10 toluene																
11 tetrachloroethylene																
12 chlorobenzene																
13 bromoform																
14 dibromochloropropane																
15 styrene																
16 m-dichlorobenzene																
17 p-dichlorobenzene																
18 o-dichlorobenzene																
19 ethylbenzene																
20 o-xylene																
21 m-xylene																
22 p-xylene																
Intensity of PFB (IBL)																
Amount PFB loaded																

RTI/ACS-SOP-712-001

January 1984

Revision 0

Standard Operating Procedure for Quantitation of Volatile Organic Compounds in Water

Authors:

Patricia A. Blau 3/15/84.
Patricia A. Blau Date

Donald A. Whitaker 3/15/84
Donald A. Whitaker Date

Approved:

CM Sparacino 3/19/84
Laboratory Manager, RTI/ACS Date

3/15/84
QA Officer, RTI/ACS Date

E Pellizzari 3/16/84
Vice-President, RTI/ACS Date

Standard Operating Procedure for the Quantitation of Volatile Organic Compounds in Water

RTI/ACS-SOP-712-001

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure describes the method used to quantitate the target compounds in the TEAM water samples that have been analyzed by GC/HECD/FID - Purge and Trap technique. The quantitation may be performed manually or by a BASIC Computer program written to facilitate the procedure. Version 1 of this program may be used when all targets give a linear response over the range of concentrations of the calibration curve. The program quantitates blank and control data, creates mean background and recovery files from this data, and quantitates and outputs water sample data in the TEAM Data Summary Sheet format. Version 2 of the program may be used when all HECD targets exhibit a non-linear response over the range of concentrations of the calibration curve (as typically observed). The quantitation for HECD targets is performed using a second order equation ($AX^2 + BX + C = 0$) while FID targets (which typically exhibit a linear response) are quantitated using a first order equation $Y = MX$. At the present time, background and recovery data are calculated manually and entered into the program which then quantitates and outputs data in the TEAM Data Summary Sheet format. The BASIC program is easily modifiable for non-routine applications.

2.0 SUMMARY OF THE METHOD

The quantitation of water samples is accomplished using the following data provided by the GC/Purge and Trap Operator(s).

1) FID and HECD integrator reports for blanks, controls and samples, with target peaks previously identified and underlined (see SOP-512-001 on Analysis of Drinking Water), and;
2) Calibration curve tables (e.g., see Figure 1).

Blank and control data for each target are averaged and mean background and recovery data generated. Sample data are quantitated from calibration data

Calibration Curve Data

Date: 3/31/83

Area response is x 10⁴

	ppb in 25 mL of water												
	Blank	G	F	E	D	C	B	A			slope	r corr.	y-inter.
Compound	0	.05	0.1	0.25	0.5	1.0	2.0	5.0					
BEN					1.18	2.42	5.24	☒					
Tol													
Ethylbenzene					1.06	2.10	4.63	13.3					
Sty					.88	1.93	4.21	11.8					
p-xylene					1.10	2.06	4.80	13.2					
VDC		4.67	8.79	23.0	49.3	104	255	744					
DCE					18.8	83.0	130	479					
MC		6.75	9.46	29.3	66.1	134	342	958					
CCl_4		7.61	12.5	33.4	77.2	149	401	1200					
TCE	0.91	7.42	18.9	36.0	76.7	165	385	1107					
CDM			6.33	9.67	24.0	56.5	141	406					
$CHBr_3$					10.2	251	61.0	174					
Perc	2.58	9.61	15.8	38.2	85.9	183	430	1277					
Cl Bz			5.51	12.8	27.6	60.6	137	398					
o-Cl_2Bz			☒	19.7	44.8	89.0	207	596					
	0	1.5	5	10	20	50	75	100					
$CHCl_3$	148	5216	14042	2637	5410	12679	17995	19620					
	0	0.3	1.0	2.0	4.0	10	15	20					
BDM		24.4	104	216	513	1314	2198	2886					

① All FID areas are obtained by multiplying attenuation x counts

② conc. = x
area = y

conc. unk. = $\frac{\text{area}}{\text{slope}}$

Figure 1. Example of a Calibration Curve Data Table.

and corrected for background and recovery. The principles remain the same whether the calculations are performed manually or by computer.

3.0 USE OF CALIBRATION DATA

The calibration curve data compiled by the GC/Purge and Trap operator(s) are subjected to linear regression or polynomial regression to determine the curve of best fit. Generally, two calibration curves are required for quantitation; however, there are cases when only one is available, (e.g., due to instrument failure) and must be used. The two sets of calibration data should bracket the analysis date of the samples being quantitated. Calibration curves are generated approximately every 10 working days, and only the samples analyzed within this time interval should be quantitated using this calibration data. This procedure minimizes errors due to minor system changes over time.

The two sets of calibration data are averaged, resulting in a mean response value at each concentration for the 10-day time interval.

3.1 For each target compound, plot the concentration versus response.

3.2 If the plot clearly appears to be linear or parabolic, perform the appropriate regression (i.e., linear or polynomic).

3.3 If the plot is ambiguous, perform both a linear and a polynomic regression.

NOTE: Historical data indicate that HECD targets exhibit a parabolic fit while FID targets exhibit linearity.

3.4 Select the most appropriate regression as the one that minimizes the absolute value of the Y intercept and maximizes the goodness of fit.

NOTE: The line of best fit is defined as one where the absolute value of the Y intercept is <50% of the lowest value of the calibration curve, and where the back-calculated values best compare with known concentrations of the calibration standard(s).

4.0 STORAGE OF DATA

All plots and quantitations, including calibration data, blank and control data, background and recovery data, and sample data, should be recorded and stored in an organized fashion to facilitate future reference. If the computer program is used for quantitation, the data should be stored on disk and a hardcopy printed. See Table 1 and Figure 2 for examples of computer print-outs for mean control and recovery data and for sample quantitation, respectively. The hard copies should be stored in binders available for this

Table 1. A. EXAMPLE OF A MEAN CONTROL DATA TABLE
B. EXAMPLE OF A MEAN RECOVERY DATA TABLE

MEAN, SD, AND CV FOR CONTROLS (IN PPB)
NORTH DAKOTA

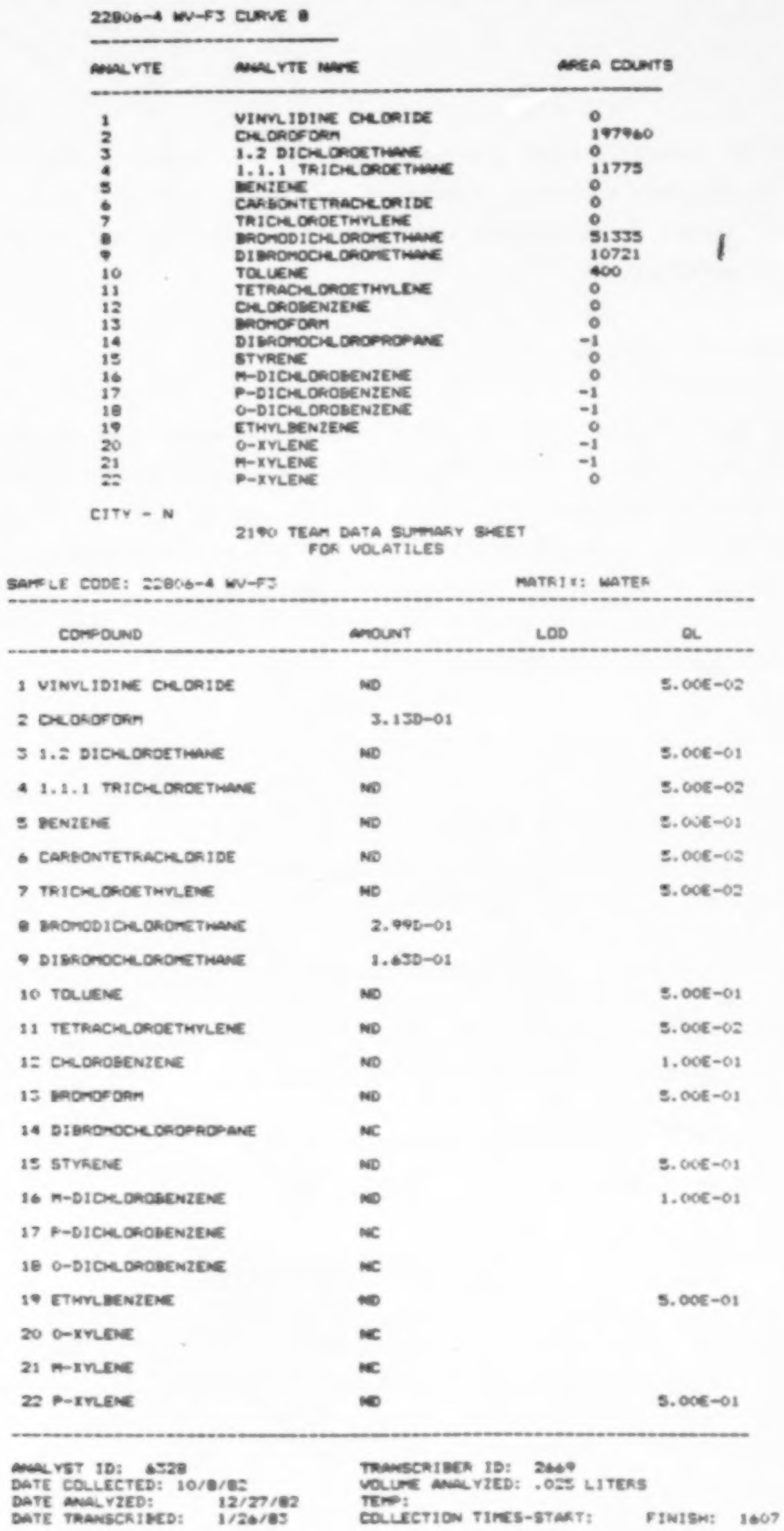
A.

ANALYTE	ANALYTE NAME	N	MEAN	SD	CV
1	VINYLIDINE CHLORIDE	4	2.19	0.09	4.2
2	CHLOROFORM	4	42.3	3.5	8.2
3	1.2 DICHLOROETHANE	4	1.36	0.19	13.9
4	1.1.1 TRICHLOROETHANE	4	2.18	0.10	4.8
5	BENZENE	4	1.81	0.06	3.6
6	CARBONTETRACHLORIDE	4	2.25	0.10	4.3
7	TRICHLOROETHYLENE	4	1.99	0.18	9.3
8	BROMODICHLOROMETHANE	4	7.07	0.73	10.3
9	DIBROMOCHLOROMETHANE	4	1.34	0.46	34.0
10	TOLUENE	4	2.17	0.29	13.4
11	TETRACHLOROETHYLENE	4	1.97	0.12	5.8
12	CHLOROBENZENE	4	1.74	0.22	12.6
13	BROMOFORM	4	1.04	0.27	26.5
14	DIBROMOCHLOROPROPANE	4	0.16	0.32	200.0
15	STYRENE	4	1.67	0.31	18.5
16	M-DICHLOROBENZENE	4	1.84	0.23	12.3
17	P-DICHLOROBENZENE	4	0.00	0.00	0.0
18	O-DICHLOROBENZENE	4	0.00	0.00	0.0
19	ETHYLBENZENE	4	1.81	0.04	2.1
20	O-XYLENE	4	0.00	0.00	0.0
21	M-XYLENE	4	0.00	0.00	0.0
22	P-XYLENE	4	1.71	0.07	4.3

MEAN RECOVERY
NORTH DAKOTA

B.

ANALYTE	ANALYTE NAME	MEAN RECOVERY
1	VINYLIDINE CHLORIDE	1.12
2	CHLOROFORM	0.91
3	1.2 DICHLOROETHANE	0.70
4	1.1.1 TRICHLOROETHANE	1.12
5	BENZENE	0.93
6	CARBONTETRACHLORIDE	1.15
7	TRICHLOROETHYLENE	1.02
8	BROMODICHLOROMETHANE	0.75
9	DIBROMOCHLOROMETHANE	0.69
10	TOLUENE	1.11
11	TETRACHLOROETHYLENE	1.01
12	CHLOROBENZENE	0.89
13	BROMOFORM	0.53
14	DIBROMOCHLOROPROPANE	0.00
15	STYRENE	0.86
16	M-DICHLOROBENZENE	0.95
17	P-DICHLOROBENZENE	0.00
18	O-DICHLOROBENZENE	0.00
19	ETHYLBENZENE	0.93
20	O-XYLENE	0.00
21	M-XYLENE	0.00
22	P-XYLENE	[illegible]



```
22806-4 WV-F3 CURVE 8
-------------------------

ANALYTE         ANALYTE NAME                         AREA COUNTS
-------------------------------------------------------------

 1              VINYLIDINE CHLORIDE                    0
 2              CHLOROFORM                             197960
 3              1.2 DICHLOROETHANE                     0
 4              1.1.1 TRICHLOROETHANE                  11775
 5              BENZENE                                0
 6              CARBONTETRACHLORIDE                    0
 7              TRICHLOROETHYLENE                      0
 8              BROMODICHLOROMETHANE                   51335
 9              DIBROMOCHLOROMETHANE                   10721
10              TOLUENE                                400
11              TETRACHLOROETHYLENE                    0
12              CHLOROBENZENE                          0
13              BROMOFORM                              0
14              DIBROMOCHLOROPROPANE                  -1
15              STYRENE                                0
16              M-DICHLOROBENZENE                      0
17              P-DICHLOROBENZENE                     -1
18              O-DICHLOROBENZENE                     -1
19              ETHYLBENZENE                           0
20              O-XYLENE                              -1
21              M-XYLENE                              -1
22              P-XYLENE                               0

CITY - N
```

```
                        2190 TEAM DATA SUMMARY SHEET
                               FOR VOLATILES

SAMPLE CODE: 22806-4 WV-F3                          MATRIX: WATER
----------------------------------------------------------------------

    COMPOUND                         AMOUNT          LOD          QL
----------------------------------------------------------------------

 1 VINYLIDINE CHLORIDE                ND                       5.00E-02

 2 CHLOROFORM                          3.13D-01

 3 1.2 DICHLOROETHANE                 ND                       5.00E-01

 4 1.1.1 TRICHLOROETHANE              ND                       5.00E-02

 5 BENZENE                            ND                       5.00E-01

 6 CARBONTETRACHLORIDE                ND                       5.00E-02

 7 TRICHLOROETHYLENE                  ND                       5.00E-02

 8 BROMODICHLOROMETHANE                2.99D-01

 9 DIBROMOCHLOROMETHANE                1.63D-01

10 TOLUENE                            ND                       5.00E-01

11 TETRACHLOROETHYLENE                ND                       5.00E-02

12 CHLOROBENZENE                      ND                       1.00E-01

13 BROMOFORM                          ND                       5.00E-01

14 DIBROMOCHLOROPROPANE               NC

15 STYRENE                            ND                       5.00E-01

16 M-DICHLOROBENZENE                  ND                       1.00E-01

17 P-DICHLOROBENZENE                  NC

18 O-DICHLOROBENZENE                  NC

19 ETHYLBENZENE                       ND                       5.00E-01

20 O-XYLENE                           NC

21 M-XYLENE                           NC

22 P-XYLENE                           ND                       5.00E-01

----------------------------------------------------------------------

ANALYST ID:  6328                  TRANSCRIBER ID:  2669
DATE COLLECTED: 10/8/82            VOLUME ANALYZED: .025 LITERS
DATE ANALYZED:       12/27/82      TEMP:
DATE TRANSCRIBED:    1/26/83       COLLECTION TIMES-START:     FINISH:  1607
```

Figure 2. Area Count Print-Out with TEAM Data Summary Sheet.

purpose and covers labeled appropriately. If calculations are performed manually, all data should be tabulated in formats similar to the computer-generated tables to facilitate interpretation. They should be stored in a bound RTI notebook.

5.0 LIMITS OF QUANTITATION

The quantifiable limit for each target compound is the minimum amount that can be quantitated using the specific instrumental conditions as outlined in SOP-512-001 (Analysis of Drinking Water), but may not exceed the lower limit set by the least concentrated calibration standard. See Table 2 for quantifiable limits for the TEAM target compounds.

6.0 FIELD BLANK AND CONTROL DATA

Field blank and control sets, exposed in the field, are used to determine the mean background and the percent recoveries of the target compounds.

6.1 Quantitation of Field Blanks

Determine the concentration of each target compound in every field blank.

6.1.1 For linear relationships,

$$\text{concentration (ppb)} = \frac{\text{area counts}}{\text{slope}}$$

6.1.2 For parabolic relationships, solve for the two roots of the quadratic equation, as follows:

$$AX^2 + BX + C = \text{Area Counts}$$

$$AX^2 + BX + (C - \text{Area Counts}) = 0$$

$$\text{Root 1} = -\frac{B}{2A} + \frac{[B^2 - 4A(C - \text{area counts})]^{1/2}}{2A}$$

$$\text{Root 2} = -\frac{B}{2A} - \frac{[B^2 - 4A(C - \text{area counts})]^{1/2}}{2A}$$

Table 2. QUANTIFIABLE LIMITS

Compound		Quantifiable Limit
01	vinylidene chloride (VDC)	5 - 02
02	chloroform ($CHCl_3$)	5 - 02
03	1,2 dichloroethane (DCE)	5 - 01
04	1,1,1 trichloroethane (MC)	5 - 02
05	benzene (BEN)	5 - 01
06	carbontetrachloride (CCl_4)	5 - 02
07	trichloroethylene (TCE)	5 - 02
08	bromodichloromethane (BDM)	1 - 01
09	dibromochloromethane (CDM)	1 - 01
10	toluene (TOL)	5 - 01
11	tetrachloroethylene (PERC)	5 - 02
12	chlorobenzene (ClBEN)	1 - 01
13	bromoform ($CHBr_3$)	5 - 01
14	dibromochloropropane (DBCP)	NC -
15	styrene (STY)	5 - 01
16	m-dichlorobenzene (mCl_2BEN)	1 - 01
17	p-dichlorobenzene (pCl_2BEN)	NC -
18	o-dichlorobenzene (oCl_2BEN)	NC -
19	ethylbenzene (EtBEN)	5 - 01
20	o-xylene (o-XYL)	NC -
21	m-xylene (m-XYL)	NC -
22	p-xylene (p-XYL)	5 - 01

6.1.2.1 Reject the negative root.

6.1.2.2 Accept the positive root as concentration (in ppb).

6.2 Calculating the Mean Background Concentration

For all the field blank data, average the concentration (in ppb) for each target compound.

6.3 Quantitation of Field Controls

Determine the concentration of each target compound in every field control.

6.3.1 For linear relationships, follow step 6.1.1.

6.3.2 For parabolic relationships, follow step 6.1.2.

6.4 Quantitation of Mean Control Concentration

6.4.1 For all the field control data, average the concentration (in ppb) for each target compound.

6.4.2 Determine the standard deviation for each target compound.

6.4.3 Determine the coefficient of variation for each target compound.

6.5 Calculating the Mean Recovery

For each target compound, determine the mean recovery using the following equation:

$$\text{mean recovery} = \frac{\text{mean control concentration - mean background concentration}}{\text{concentration spiked into the controls}}$$

7.0 SAMPLE QUANTITATION

7.1 For each target compound that exhibits a linear response, perform the following calculation.

$$\text{uncorrected conc. (in ppb)} = \frac{\text{area counts}}{\text{slope}} - \text{mean background}$$

7.2 If the uncorrected concentration is less than the quantifiable limit, report the target compound as "Not Detected", otherwise correct for recovery using the following calculation:

$$\text{corrected conc. (in ppb)} = \frac{\text{uncorrected conc.}}{\text{mean recovery}}$$

7.3 For each target compound that exhibits a parabolic response, perform the following calculations.

7.3.1 Determine the roots of the quadratic equation:

$$\text{Roots} = -\frac{B}{2A} \pm \frac{[B^2 - 4A(C - \text{area counts})^{1/2}}{2A}$$

7.3.2 Subtract the mean background from the positive root to determine the uncorrected concentration (in ppb).

7.3.3 If the uncorrected concentration is less than the quantifiable limit, report the target compound as "Not Detected", otherwise correct for recovery using the following calculation:

$$\text{corrected conc. (in ppb)} = \frac{\text{uncorrected conc.}}{\text{mean recovery}}$$

8.0 DATA REPORT FORMS

In order the facilitate the statistical analysis of the data, the quantities of target compounds detected are arranged in a specific format for further manipulation. If the computer program mentioned earlier is utilized, it correctly formats the data automatically; however, if calculations are done by hand, the data must be transcribed to data summary sheets as shown in Figure 3. All information must be filled in with the exception of the "temperature" and "start collection time" (see the example).

8.1 Directions for Filling Out Data Summary Sheet

8.1.1 Obtain one sheet for each sample.

8.1.2 Write the sample code in the blocks provided.

NOTE: Boxes 1-6 are for the respondent number and boxes 7-8 are for the matrix code (WV). Boxes 9-12 are for the sample type and number (i.e., F, D, FB1, FB2, etc.) Boxes 1-7 and 9 must always be filled in.

8.1.3 Enter the sample quantities.

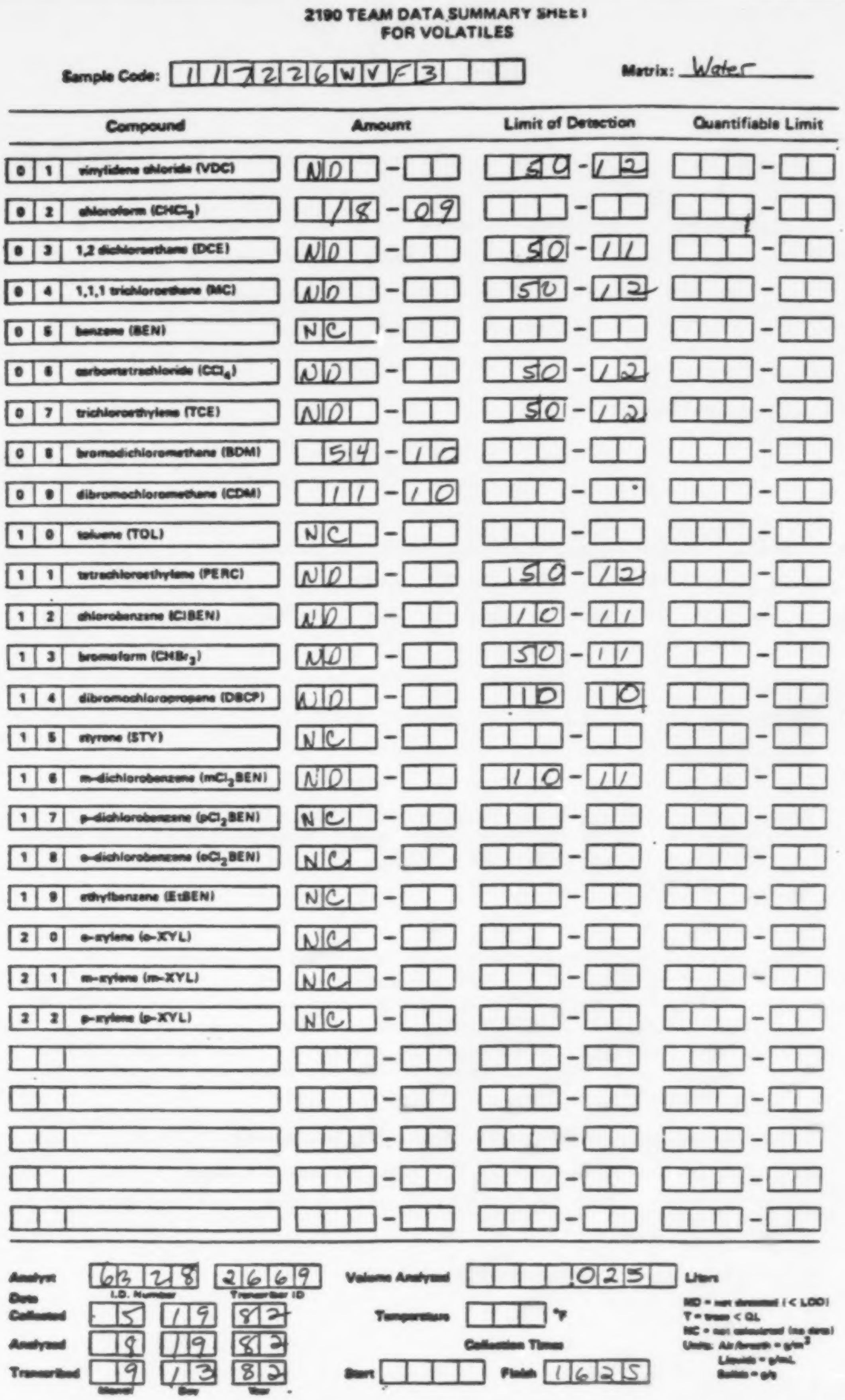
NOTE: The entries are set-up in scientific notation. The first three boxes are for the amount and the following two boxes for the exponent. The units for water are ppb (ng/mL). All ND (not detected) or NC (not calculated) entries are left-hand justified. A ND notation requires a value entered in the Quantifiable Limit column. Numerical values are right-hand justified.

8.1.4 Enter the Analyst's RTI identification number.

8.1.5 Enter the Transcriber's RTI identification number.

8.1.6 Enter the date collected, the data analyzed, and the date transcribed.

8.1.7 Enter the volume analyzed in liters.



2190 TEAM DATA SUMMARY SHEET
FOR VOLATILES

Sample Code: 1 1 7 2 2 6 W V F 3

Matrix: Water

	Compound	Amount	Limit of Detection	Quantifiable Limit
0 1	vinylidene chloride (VDC)	ND -	50 - 12	-
0 2	chloroform ($CHCl_3$)	18 - 09	-	-
0 3	1,2 dichloroethane (DCE)	ND -	50 - 11	-
0 4	1,1,1 trichloroethane (MC)	ND -	50 - 12	-
0 5	benzene (BEN)	NC -	-	-
0 6	carbontetrachloride (CCl_4)	ND -	50 - 12	-
0 7	trichloroethylene (TCE)	ND -	50 - 12	-
0 8	bromodichloromethane (BDM)	54 - 10	-	-
0 9	dibromochloromethane (CDM)	11 - 10	-	-
1 0	toluene (TOL)	NC -	-	-
1 1	tetrachloroethylene (PERC)	ND -	50 - 12	-
1 2	chlorobenzene (ClBEN)	ND -	10 - 11	-
1 3	bromoform ($CHBr_3$)	ND -	50 - 11	-
1 4	dibromochloropropane (DBCP)	ND -	10 10	-
1 5	styrene (STY)	NC -	-	-
1 6	m-dichlorobenzene (mCl_2BEN)	ND -	10 - 11	-
1 7	p-dichlorobenzene (pCl_2BEN)	NC -	-	-
1 8	o-dichlorobenzene (oCl_2BEN)	NC -	-	-
1 9	ethylbenzene (EtBEN)	NC -	-	-
2 0	o-xylene (o-XYL)	NC -	-	-
2 1	m-xylene (m-XYL)	NC -	-	-
2 2	p-xylene (p-XYL)	NC -	-	-
		-	-	-
		-	-	-
		-	-	-
		-	-	-
		-	-	-

Analyst: 6328 (I.D. Number) 2669 (Transcriber ID)

Volume Analyzed: .025 Liters

Date	Month	Day	Year
Collected	5	19	82
Analyzed	8	19	82
Transcribed	9	13	82

Temperature: °F

Collection Times

Start:

Finish: 1625

ND = not detected (< LOD)
T = trace < QL
NC = not calculated (no data)
Units: Air/breath = g/m³
Liquids = g/mL
Solids = g/g

Figure 3. Data Summary Sheet.

8.1.8 Enter the sample time of collection.

NOTE: The above information is obtained from the sample collection chain of custody sheets for each sample.

9.0 QUALITY ASSURANCE CHECK

Twenty-five percent of the samples quantitated must be checked for correctness by another individual.

9.1 Instructions for QA Check

9.1.1 Obtain from the transcriber the notebook containing the integrator output for the samples and standards, and the TEAM Data Summary sheets.

9.1.2 Randomly select 25% of the samples and check to ensure that the target compounds were correctly identified and underlined.

9.1.3 Initial each sample's integrator output checked.

9.1.4 Prepare a list of any errors found.

9.1.5 Check of Data Entered into the Computer

9.1.5.1 Compare the area counts on the integrator output with the computer printout of area counts (see Figure 2).

9.1.5.2 Check to ensure that the sample code, dates, times and ID numbers are correct.

9.1.5.3 Prepare a list of any errors found.

9.1.6 Check of Data Calculated Manually

9.1.6.1 Check all calculations to ensure that the values reported are correct.

9.1.6.2 Repeat steps 9.1.5.2 and 9.1.5.3 above.

9.2 Correction of Errors

9.2.1 Submit to the transcriber the list of errors found (if any).

9.2.2 The transcriber will make all necessary corrections and initial where appropriate (i.e., on the integrator output or in the bound RTI notebook) that the errors were corrected.

10.0 PREPARATION FOR BATCHING

The data summary sheets must be submitted to CCA in groups called batches.

10.1 Obtain a copy of the sampling schedule for the sampling trip.

10.2 Make photocopies of the data summary sheets.

10.3 Put the data summary sheets in numerical order.

10.4 Check off on the schedule each sample for which there is a data summary sheet.

10.5 Submit the data summary sheets to the individual responsible for batching.

10.6 After all the data summary sheets have been submitted for batching, check the sampling schedule and determine if any data is missing.

 10.6.1 If there is data missing, determine whether data is available or whether the sample was lost or broken.

 NOTE: If a sample was scheduled to be collected and was either not collected, lost or broken, all the compounds for that sample will be reported NC.

RTI/ACS-SOP-790-001
January 1984
Revision 0

Standard Operating Procedure for the Preparation and Submission of Data Summary Sheets to the Center for Computer Applications/Data Entry (CCA/DE)

Author:

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Standard Operating Procedure for the Preparation and Submission of Data Summary Sheets to the Center for Computer Applications/Data Entry (CCA/DE)

RTI/ACS-SOP-790-001

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure describes a process called batching which is a method used to organize and group data summary forms that are to be processed by CCA/DE. The same procedure could be applied to any data that is presented in a format that conforms to the specifications of the Data Entry program to be used.

2.0 PRINCIPLE

The final results of the analysis of each field sample are reported on a data summary sheet. A data summary sheet has been devised for each of the groups of chemicals that have been chosen as the target compounds for the project (see Appendix, pages 1-3). Before these data summary sheets can be processed in CCA/DE, they must be put into groups called batches.

3.0 PREPARATION

3.1 Site Batching Notebook

Each batch that is assembled has a corresponding batching sheet (see Appendix, page 4). This batching sheet serves as a record of the contents of the batch. A copy of each completed batching sheet will be kept on record in the Site Batching Notebook.

3.1.1 Request from the lab supervisor a list of the sample matrices and the number of samples of each type that were collected.

3.1.2 Use loose leaf index dividers to make a section in a three-ring binder for each matrix.

NOTE: If a sampling site has more than one distinct area where sample collection took place, assign separate sections for each of the areas for each matrix.

3.2 Batching Labels

Each data summary sheet in a batch is given a document id number that is independent of any characteristics of the sample. The document id numbers are printed in pairs on stickers and are available upon request from CCA.

3.2.1 From the list of samples that will be submitted for batching, determine the number of pairs of document stickers needed.

3.2.2 Request document stickers from the CCA member responsible for this task.

NOTE: On subsequent requests for ID stickers, inform the CCA member of the number on the last sticker of the previous printing.

4.0 PROCEDURE

4.1 Batching

The data summary sheets that constitute a batch will have all of the following in common: 1) site, 2) city or town (if applicable), 3) matrix, and 4) Tenax batch (for air and breath samples). Each batch is identified by a five digit number. The first digit corresponds to site, the second to media, the third to chemical type, and the fourth and fifth to the actual number of the batch (see Appendix, page 5). For example, a batch with the number 12158 would be the 58th batch of water samples collected in New Jersey and analyzed for volatile organic compounds.

4.1.1 Briefly look over each sheet of the group submitted to make sure they are complete and legible.

NOTE: Sheets will be checked for correctness before being submitted for batching.

4.1.2 Separate sheets into groups such that each group has the following in common: 1) site, 2) city or town (if applicable), 3) matrix and 4) Tenax batch (if applicable).

NOTE: In many cases, the entire group of sheets submitted will have all of these characteristics in common and will not need to be separated.

4.1.3 Put the data sheets in each group in numerical order.

4.1.4 Fill out batching sheet (Refer to Appendix, page 6).

4.1.4.1 Beginning with one of the groups in 3.2.2, determine the correct batch number and write it in on the batching sheet.

4.1.4.2 Put date and initials in the designated space.

4.1.4.3 In the upper right corner of the sheet, record the site, city (if applicable), matrix and Tenax batch (if applicable).

4.1.4.4 Using the paired document id stickers, place one of the first pair on the first data summary sheet and the second on the batching sheet in the designated space. Repeat with the second sheet and so on until all the sheets in the group or the first 25 are batched.

4.1.4.5 Write in the batch count.

4.1.4.6 Write the transcriber ID number, which appears on the bottom of the data summary sheet, in the designated spaces.

NOTE: If one individual transcribed the entire batch, the ID number need only appear in the first space.

4.1.5 Secure the batch with a binder clip.

4.1.6 If more batches need to be assembled, repeat from 4.1.4.

4.1.7 Photocopy

4.1.7.1 Make a photocopy of each batch.

4.1.7.2 Check the batch count of the originals and make sure they are in numerical order.

4.1.7.3 Check the batch count of the copies and make sure there is a copy for each original.

4.1.7.3.1 Secure each batch with a binder clip.

4.2 Submission to CCA/DE

4.2.1 Deliver the completed batches to the CCA member responsible for this task.

5.0 RECORDKEEPING

5.1 Data Entry Batches

5.1.1 Taking one batch at a time, separate the batching sheet from the data summary sheets.

5.1.2 Write the batch number on the first sheet of the batch.

5.1.3 Store the copies separated by site and matrix and in numerical order.

5.2 Batching Sheets

5.2.1 File the batching sheets into the appropriate sections of the Site Batching Notebook.

5.2.2 Make a list for each media of all the batches assembled. Keep these lists at the front of the Site Batching Notebook.

NOTE: These lists will serve as a record of what batch numbers have been used.

APPENDIX

1

2190 TEAM DATA SUMMARY SHEET
FOR VOLATILES

Sample Code: ☐☐☐☐☐☐☐☐☐☐☐☐☐ Matrix: ____________

Compound		Amount	Limit of Detection	Quantifiable Limit
0 1	vinylidene chloride (VDC)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
0 2	chloroform ($CHCl_3$)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
0 3	1,2 dichloroethane (DCE)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
0 4	1,1,1 trichloroethane (MC)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
0 5	benzene (BEN)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
0 6	carbontetrachloride (CCl_4)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
0 7	trichloroethylene (TCE)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
0 8	bromodichloromethane (BDM)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
0 9	dibromochloromethane (CDM)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
1 0	toluene (TOL)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
1 1	tetrachloroethylene (PERC)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
1 2	chlorobenzene (ClBEN)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
1 3	bromoform ($CHBr_3$)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
1 4	dibromochloropropane (DBCP)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
1 5	styrene (STY)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
1 6	m-dichlorobenzene (mCl_2BEN)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
1 7	p-dichlorobenzene (pCl_2BEN)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
1 8	o-dichlorobenzene (oCl_2BEN)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
1 9	ethylbenzene (EtBEN)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
2 0	o-xylene (o-XYL)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
2 1	m-xylene (m-XYL)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
2 2	p-xylene (p-XYL)	☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
☐☐		☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
☐☐		☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
☐☐		☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
☐☐		☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐
☐☐		☐☐☐ – ☐☐	☐☐☐ – ☐☐	☐☐☐ – ☐☐

Analyst ☐☐☐☐ ☐☐☐☐
I.D. Number Transcriber ID

Date	Month	Day	Year
Collected	☐☐	☐☐	☐☐
Analyzed	☐☐	☐☐	☐☐
Transcribed	☐☐	☐☐	☐☐

Volume Analyzed ☐☐☐☐☐☐☐☐ Liters

Temperature ☐☐☐ °F

Collection Times

Start ☐☐☐☐ Finish ☐☐☐☐

ND = not detected (< LOD)
T = trace < QL
NC = not calculated (no data)
Units: Air/breath = g/m^3
Liquids = g/mL
Solids = g/g

2190 TEAM DATA SUMMARY SHEET
FOR PESTICIDES

Sample Code: ☐☐☐☐☐☐☐☐☐☐☐☐☐ Matrix: ____________

Compound			Amount	Limit of Detection	Quantifiable Limit
4	1	HCB	☐☐☐–☐☐	☐☐☐–☐☐	☐☐☐–☐☐
4	2	α-BHC	☐☐☐–☐☐	☐☐☐–☐☐	☐☐☐–☐☐
4	3	γ-BHC	☐☐☐–☐☐	☐☐☐–☐☐	☐☐☐–☐☐
4	4	β-BHC	☐☐☐–☐☐	☐☐☐–☐☐	☐☐☐–☐☐
4	5	heptachlor epoxide	☐☐☐–☐☐	☐☐☐–☐☐	☐☐☐–☐☐
4	6	t-nonachlor	☐☐☐–☐☐	☐☐☐–☐☐	☐☐☐–☐☐
4	7	p,p′-DDE	☐☐☐–☐☐	☐☐☐–☐☐	☐☐☐–☐☐
4	8	p,p′-DDD	☐☐☐–☐☐	☐☐☐–☐☐	☐☐☐–☐☐
4	9	p,p′-DDT	☐☐☐–☐☐	☐☐☐–☐☐	☐☐☐–☐☐
5	0	heptachlor	☐☐☐–☐☐	☐☐☐–☐☐	☐☐☐–☐☐
5	1	chlordane	☐☐☐–☐☐	☐☐☐–☐☐	☐☐☐–☐☐
5	2	oxychlordane	☐☐☐–☐☐	☐☐☐–☐☐	☐☐☐–☐☐
5	3	PCBs	☐☐☐–☐☐	☐☐☐–☐☐	☐☐☐–☐☐
☐	☐		☐☐☐–☐☐	☐☐☐–☐☐	☐☐☐–☐☐
☐	☐		☐☐☐–☐☐	☐☐☐–☐☐	☐☐☐–☐☐
☐	☐		☐☐☐–☐☐	☐☐☐–☐☐	☐☐☐–☐☐
☐	☐		☐☐☐–☐☐	☐☐☐–☐☐	☐☐☐–☐☐
☐	☐		☐☐☐–☐☐	☐☐☐–☐☐	☐☐☐–☐☐
☐	☐		☐☐☐–☐☐	☐☐☐–☐☐	☐☐☐–☐☐

Analyst ☐☐☐☐ ☐☐☐☐
I.D. Number Transcriber ID

Date
Collected ☐☐–☐☐–☐☐
Analyzed ☐☐ ☐☐ ☐☐
Transcribed ☐☐ ☐☐ ☐☐
Month Day Year

Volume Analyzed ☐☐☐☐.☐☐☐☐ Liters

Temperature ☐☐☐ °F

Collection Times
Start ☐☐☐☐ Finish ☐☐☐☐

ND = not detected (< LOD)
T = trace < QL
NC = not calculated (no data)
Units: Air/breath = g/m^3
Liquids = g/mL
Solids = g/g

3

2190 TEAM DATA SUMMARY SHEET
FOR METALS

Sample Code: ☐☐☐☐☐☐☐☐☐☐☐☐☐ **Matrix:** ______________

Compound			Amount	Limit of Detection	Quantifiable Limit
7	1	arsenic (Ar)	–	–	–
7	2	lead (Pb)	–	–	–
7	3	cadmium (Cd)	–	–	–
			–	–	–
			–	–	–
			–	–	–
			–	–	–
			–	–	–
			–	–	–
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			–	–	–
			–	–	–
			–	–	–

Analyst ☐☐☐☐ (I.D. Number) ☐☐☐☐ (Transcriber ID)

Date

Collected ☐☐ ☐☐ ☐☐

Analyzed ☐☐ ☐☐ ☐☐

Transcribed ☐☐ ☐☐ ☐☐

Month Day Year

Volume Analyzed ☐☐☐☐☐☐☐☐ Liters

Temperature ☐☐☐ °F

Collection Times

Start ☐☐☐☐ **Finish** ☐☐☐☐

ND = not detected (< LOD)
T = trace < QL
NC = not calculated (no data)
Units: Air/breath = g/m^3
Liquids = g/mL
Solids = g/g

BATCH NUMBER ______

BATCH COUNT __

	Date	Initials
Batched (ASD)		
Data Entry (CAC)		
Received		
1st Keying		
Verification		
Checked Out		
Received (ASD)		

	STICKER ID	Transcriber ID
1		
2		
3		
4		
5		
6		
7		
8		

	STICKER ID	Transcriber ID
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		
20		
21		
22		
23		
24		
25		

Batch Numbers

5-digit code

First digit - Site:
1. New Jersey
2. North Carolina
3. Louisiana
4. Texas
5. New York
6. North Dakota
7. California
8.
9. Special Studies

Second digit - Media:
1. Air/Breath
2. Water
3. Urine, Blood, Hair, Mother's Milk
4. Food

Third digit - Chemical Type:
1. Volatiles
2. Pesticides/PCBs
3. Metals

Fourth and Fifth digits - Batch #

[illegible]

BATCH NUMBER 11122

	Date	Initials
Batched (ASD)	4/29/83	KaB
Data Entry (CAC)		
Received		
1st Keying		
Verification		
Checked Out		
Received (ASD)		

	STICKER ID	Transcriber ID
1	TEAM 04305-9	2343
2	TEAM 04306-7	
3	TEAM 04307-5	
4	TEAM 04308-3	
5	TEAM 04309-1	
6	TEAM 04310-9	
7	TEAM 04311-7	
8	TEAM 04312-5	

BATCH COUNT 20

	STICKER ID	Transcriber ID
9	TEAM 04313-3	
10	TEAM 04314-1	
11	TEAM 04315-8	
12	TEAM 04316-6	
13	TEAM 04317-4	
14	TEAM 04318-2	
15	TEAM 04319-0	
16	TEAM 04320-8	
17	TEAM 04321-6	
18	TEAM 04322-4	
19	TEAM 04323-2	
20	TEAM 04324-0	
21		
22		
23		
24		
25		

RTI/ACS-SOP-810-001

January 1984

Revision 0

Standard Operating Procedure for the Preparation and Handling of QA Audit Samples on Tenax for GC/MS Analysis

Author:

Doris J. Smith 2-7-84
Doris J. Smith
Date

Approved:

Robert W Handy 2/7/84
Robert W. Handy
ACS QA Officer
Date

E Pellizzari 2/7/84
E. D. Pellizzari
ACS Vice-President
Date

Standard Operating Procedure for the Preparation and Handling of QA Audit Samples on Tenax for GC/MS Analysis

RTI/ACS-SOP-810-001

1.0 PRINCIPLE

Tenax cartridges are spiked with known amounts of target compounds, disguised by labeling as field samples and then sent to either the MS laboratory or a QA/QC laboratory for analysis.

2.0 MATERIALS/SUPPLIES

Clean Tenax cartridges sealed in culture tubes
Chain-of-Custody Sheets
Labels
Sampling Schedule

3.0 PROCEDURE

3.1 Procuring Clean Tenax Cartridges

Send a memo to the Project Director requesting a number of clean Tenax cartridges, preferably a week before field sampling begins. The number may vary, but is typically ten - 3 for EPA, 2 for the QA/QC lab and 5 for RTI.

3.2 Temporary Labeling

Label each cartridge with a descriptive code (QA-IN for Quality Assurance - Indoor Air, for example). As an example, the labels for a group of 10 cartridges follow:

QA-IN-11 (QA Lab)
QA-IN-12 (QA Lab)
QA-IN-13 (RTI)
QA-IN-14 (RTI)
QA-IN-15 (RTI)
QA-IN-16 (RTI)
QA-IN-17 (RTI)
QA-IN-18 (RTI)
QA-IN-19 (RTI)
QA-IN-20 (RTI)

The next series would start with QA-IN-21. A chain-of-custody sheet (Figure 1) is filled out for each cartridge. The sheet is filled out as shown in Figure 2, except for the changed sample code entry (see Section 3.5.1.3).

3.3 Delivery of Cartridges to EPA

Deliver the cartridges and chain-of-custody sheets to the QA contact person at EPA. The cartridges are loaded with known amounts of target compounds at the discretion of EPA.

3.4 Return of Cartridges to RTI

When loading of the cartridges with selected target compounds is complete, the cartridges assigned to RTI and the QA/QC lab and their custody sheets are returned to RTI.

3.5 Relabeling the Cartridges

The cartridges are now ready to be relabeled as field samples and added to the existing field samples.

3.5.1 Relabel all audit samples as duplicates of field samples or duplicates of QA field samples if audit material is sent to a QA lab.

3.5.1.1 Identify eligible samples codes for the audit samples.

NOTE: There must be no duplication in the use of sample identification numbers and the label used must be similar in all respects to labels used for the samples in the field.

3.5.1.2 Select sample inditification code numbers for the audit samples. Make assignments uniformily as possible over the total sample collection with respect to sampling site and time.

NOTE: For example, if an equal number of samples were collected at two different sites 1 and 2, select an equal number of site 1 and 2 identification codes for the audit samples. If the collection period was 4 weeks and 4 audit samples need to be labelled, select a sample code number used in month 1, 2, 3, and 4.

3.5.1.3 Note the selected sample number on the corresponding initial chain-of-custody sheet (see Figure 2). Retain initial custody sheets in QA File.

3.5.1.4 Remove the temporary label from the culture tube containing the cartridge and apply the new one.

3.5.2 Fill out a new chain-of-custody sheet identifying the specimen with its new sample number (see Figure 3). Make no reference to the initial custody sheet or temporary identification number.

NOTE: Line items (e.g., sample exposed, collected, QA'd, shipped, received) should be forged to resemble custody sheets for actual samples.

3.6 Submitting Cartridges for GC/MS Analysis

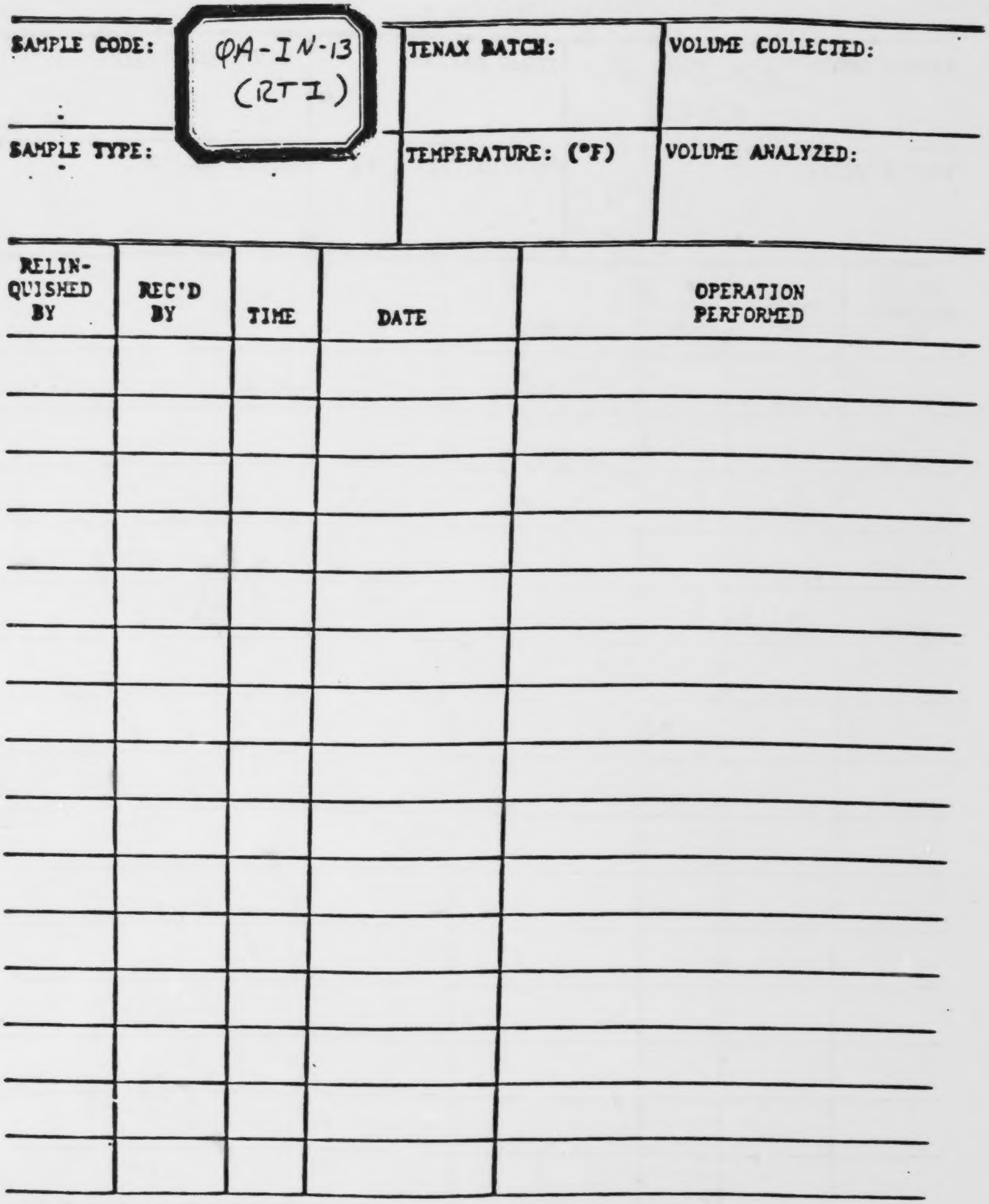
3.6.1 Store the audit samples with the collected field samples and merge the forged custody sheets with those for the authentic samples.

3.6.2 Inform the individual in charge of data processing, the project leader, and the QA Officer which study codes were used on the audit samples. This communication should be in the form of a written memo.

3.6.3 When the raw analytical data arrives from GC/MS, the QA Officer or designate shall review the output first to remove the audit results.

CAUTION

Failure to remove the audit data will result in this information being entered to the file as actual duplicate analysis.

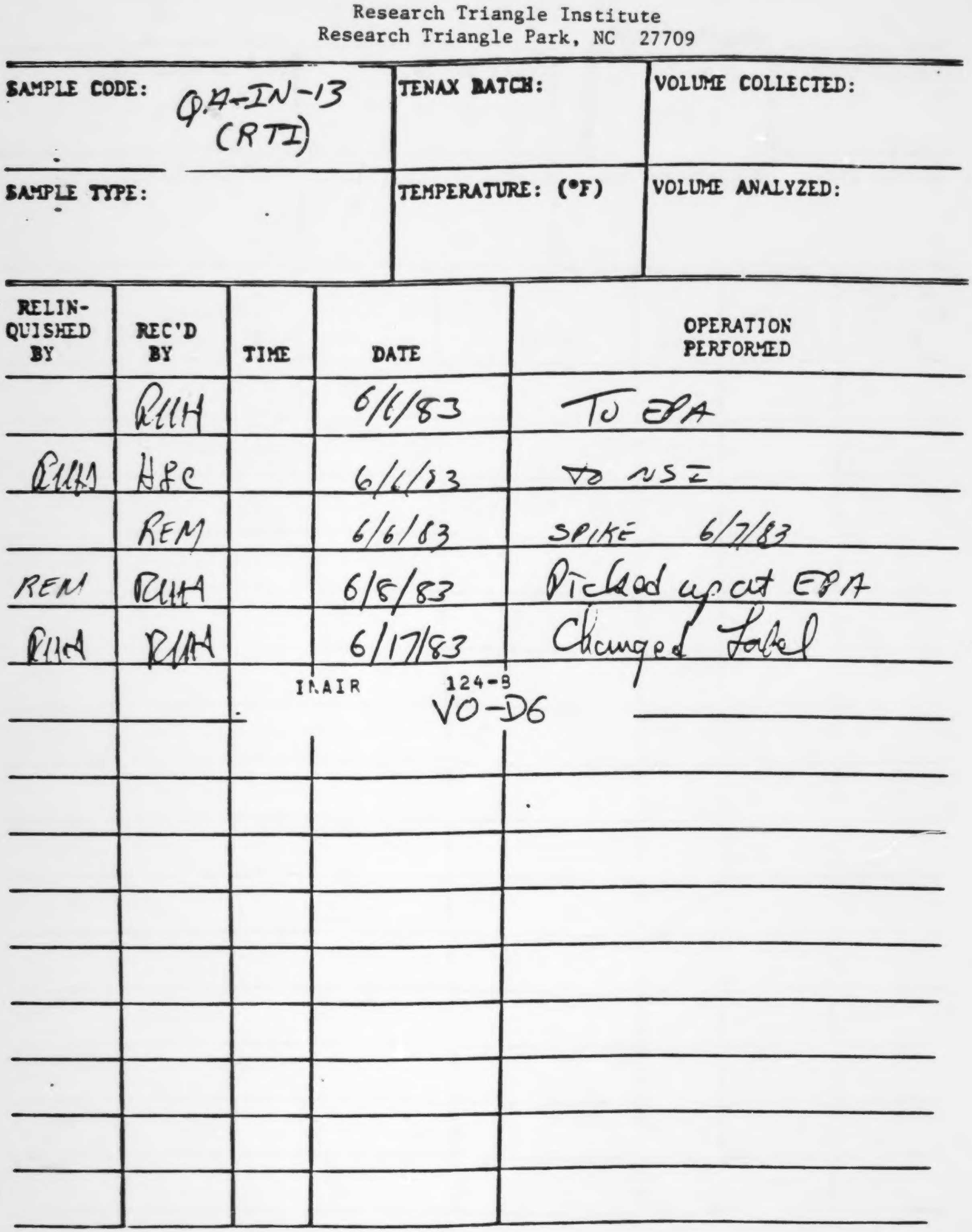


Research Triangle Institute, Analytical and Chemical Sciences
Research Triangle Park, NC 27709

SAMPLE CODE: QA-IN-13 (RTI)	**TENAX BATCH:**	**VOLUME COLLECTED:**
SAMPLE TYPE:	**TEMPERATURE: (°F)**	**VOLUME ANALYZED:**

RELINQUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED

Figure 1. An Initial Chain-of-Custody Sheet for QA Audit Samples



Research Triangle Institute
Research Triangle Park, NC 27709

SAMPLE CODE: QA-IN-13 (RTI)	**TENAX BATCH:**	**VOLUME COLLECTED:**
SAMPLE TYPE:	**TEMPERATURE: (°F)**	**VOLUME ANALYZED:**

RELIN-QUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	RHH		6/6/83	TO EPA
RHH	HJC		6/6/83	TO NSI
	REM		6/6/83	SPIKE 6/7/83
REM	RHH		6/8/83	Picked up at EPA
RHH	RHH		6/17/83	Changed label

INAIR 124-B

VO-D6

Figure 2. A Completed Initial Chain-of-Custody Sheet for QA Audit Samples

CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute
Research Triangle Park, NC 27709

SAMPLE CODE: INAIR 124-B VO-D6	**TENAX BATCH:** 64	**VOLUME COLLECTED:** 20.8L
SAMPLE TYPE: tex	**TEMPERATURE: (°F)** 71°	**VOLUME ANALYZED:**

RELIN-QUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	JTK	920	5/25/83	SE
JTK	JTK	1730	5/26/83	SC
JTK	JDL		5/25	QA
JDL	JTK		5/26/83	to RTI
JK	PAB		MAY 27 1983	RECEIVED AT RTI
PAB				

Figure 3. A Final Chain-of-Custody Sheet for QA Audit Samples

RTI/ACS-SOP-812-001

February 1984

Revision 0

Standard Operating Procedure for Conducting a QA System Audit of Sample and Data Collection in the Field

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Approved:

E Pellizzari

Edo D. Pellizzari
ACS Vice-President

Standard Operating Procedure for
Conducting a QA System Audit of Sample
and Data Collection in the Field
RTI/ACS-SOP-812-001

1.0 INTRODUCTION

The major objectives of a sample collection system audit are (1) to establish adherence to Standard Operating Procedures (SOPs), (2) to identify potential problem areas, (3) to recommend or evaluate modifications in routine operations to improve the efficiency and/or performance quality of specific field activities, and (4) to provide an objective assessment of overall quality of sample collection.

The value of the audit in assessing the impact of sample collection on data quality depends on the scope of the audit and the complexity of the study. To the extent possible, this concern should be addressed in the audit report to project management.

2.0 APPLICATION

The audit procedures described in this SOP have been designed for the field sampling phase of the TEAM study. Since many of the audit procedures are general in nature, this SOP should be applicable to several different projects with only minor revisions.

There are basically three different levels at which the audit may be conducted.

	Length of Time (Incl. travel)	Condition
1.	1 Day	A short audit at a nearby site. The sample collection is performed by one team.
2.	3-4 Days	A more complete audit at a more distant site. The sample collection is performed by more than one team.
3.	5-7 Days	An extended audit at a distant site. The sample collection is performed by more than one team which is replaced by other sampling personnel during the audit visit.

3.0 SHORT AUDIT

This audit shall be conducted by the QA Officer or his/her designate.

3.1 Administer the Audit Checklist (Appendix A) to the field staff.

NOTE: The responses should be given by the Site Administrator. However, everyone should feel free to contribute information or raise questions. If appropriate, such participation should be encouraged.

3.2 Observe the sampling team during each sample collection visit. Assess adherence to SOPs.

3.2.1 Carry an RTI indentification badge.

3.2.2 Explain you are there to make sure everything is going all right. Be positive. Do not give the impression that you are there because something may be wrong.

3.2.3 Do not take notes during the visit.

3.2.4 Observe all collection operations and note location of fixed air sampler, if scheduled.

3.2.5 Witness the administration of the 24 hour screener.

3.2.6 Any discussion of sampling operation with the team should wait until the visit is completed and everyone has returned to the van.

3.3 Inspect the workroom and van and assess adherence to SOPs.

3.4 Review Sampling Protocol/Chain-of-Custody sheets and assess adherence to SOP.

3.5 A written report of the audit results shall be prepared and submitted to the Task Leader and Project Director within 5 working days from return to RTI.

NOTE: Findings which require possible action should be communicated by telephone or verbally on return to RTI.

4.0 NORMAL AUDIT

This audit shall be conducted by the QA Officer or his/her designate.

4.1 Administer Audit Checklist (Appendix A) to the field staff. See Section 3.1.

4.2 Observe the sampling team during each sample collection visit. Assess adherence to SOPs. See Section 3.2.

NOTE: This activity will require observing all three visits once for the 2 or 3 sampling teams.

4.3 Inspect the workroom and vans and assess adherence to SOPs.

4.4 Review Sampling Protocol/Chain-of-Custody sheets and assess adherence to SOP.

4.5 A written report of the audit results shall be prepared and submitted to the Task Leader and Project Director. See Section 3.5.

5.0 EXTENDED AUDIT

This audit shall be conducted by the QA Officer or his/her designate. Since this audit will normally be performed during the time when the sampling teams are relieved by different sampling personnel, the following procedures shall be carried out with both groups.

5.1 Administer the Audit Checklist (Appendix A) to the field staff. See Section 3.1.

5.2 Observe the sampling team during each sample collection visit. Assess adherence to SOPs. See Section 4.2.

5.3 Inspect the workroom and vans and assess adherence to SOPs.

5.4 Review Sampling Protocol/Chain-of-Custody sheets and assess adherence to SOP.

5.5 Interview selected participants.

5.5.1 Randomly select one of the first twenty participants, one of the second twenty, etc. and conduct a brief telephone interview with these individuals.

5.5.2 This survey shall be performed no later than two weeks after the completion of sample collection.

NOTE: The interview may be conducted either at the sampling site or at RTI.

5.5.3 The discussion shall include but not be limited to the following issues:

5.5.3.1 Confirmation of participation

5.5.3.2 Confirmation of selected questionnaire data such as occupation, length of time at current residence, etc.

5.5.3.3 Impression made by the sampling team and interviewers with respect to appearance, attitude, and timeliness.

5.5.3.4 Interviewer and sampling team explanation of nature of sampling effort and participant burden involved.

5.5.3.5 Incentive payment.

5.6 Interview Interviewers

5.6.1 During the course of the audit visit, all of the interviewers used to enlist study participants shall be interviewed.

5.6.2 The discussion shall include but not be limited to the following issues.

5.6.2.1 Training/Orientation session prior to field work.

5.6.2.2 Mechanism for communicating with the Survey Director.

5.6.2.3 Comprehension of objectives and nature of study.

5.6.2.4 Interaction with potential participants.

5.6.2.5 Interaction with sampling teams during the collection phase of study.

5.7 Interview Sampling Staff

The only purpose of these discussions is to establish the success achieved during the training and orientation of each staff member and the impact on the quality and completion of sample collection. Therefore, all written reports of this evaluation shall refer to the individuals involved by code.

5.7.1 Each member of the sampling team(s) shall be interviewed individually.

5.7.2 If any sample collectors are not present or available at the site, they shall be interviewed on their return to RTI.

5.7.3 The discussion shall include but not be limited to the following issues:

5.7.3.1 Participation in sampling activities.

5.7.3.2 Comprehension of SOPs

5.7.3.3 Adherence to SOP

5.8 A written report of the audit results shall be prepared and submitted to the Task Leader and Project Director. See Section 3.5.

APPENDIX A

Audit Checklist

SYSTEMS AUDIT CHECKLIST FOR FIELD SAMPLE COLLECTION

1.0 INTERVIEWER APPOINTMENT SCHEDULE

1.1 Are morning and afternoon/evening appointments scheduled with sufficient time between sampling visits?

1.2 Are entries legible and understandable?

1.3 Are there incidences of inconsistency between the date and the day of the week?

1.4 Is there misuse (or nonuse) of the terms street, road, avenue, etc.? For example, the entry "704 Maple" is not sufficient; the entry "704 Maple St." is not useful if there is only a Maple Ave. and/or Maple Road but no Maple St.

1.5 When dealing with, a multifamily unit, is the description of participants residence clear and sufficient?

1.6 In general, how far ahead were sampling appointments made?

1.7 Comment on specific deficiencies of the interviewer appointment schedule and/or recommend measures which would improve the manner in which appointment scheduling is carried out.

2.0 SAMPLE COLLECTION - GENERAL

2.1 Has the participant received from the interviewer a true understanding of the study and what is expected of him/her?

2.2 Do sampling personnel arrive on time at the participant's residence?

2.3 Are sampling personnel suitably dressed and do they interact with participant in a professional manner?

3.0 SAMPLE COLLECTION METHODS - AIR

3.1 Are air collections being performed according to the approved SOP?

3.2 Comment on samples not collected due to pump failure; tubing separated from pump, etc.

3.3 Are fixed air samples being positioned in reasonable locations?

3.4 Comment on treatment of quality control samples when scheduled for exposure.

3.5 Identify specific deficiencies; recommend improvements.

4.0 SAMPLE COLLECTION METHODS - BREATH

4.1 Are breath collections being performed according to the approved SOP?

4.2 Describe purging of exhale air bags prior to reuse.

4.3 Indicate volume of methanol maintained at study site, where the solvent is being stored and verify that bottles containing this material are clearly identified as such.

4.4 Comment on treatment of quality control samples when scheduled for exposure.

4.5 Identify specific deficiencies; recommend improvements.

5.0 SAMPLE COLLECTION METHODS - WATER

5.1 Are water collections being performed according to the approved SOP?

5.2 Comment on time/amount of water run from tap before sample colleciton.

5.3 Comment on vapor space noted in samples.

5.4 Is there a cooler for immediate chilling of sample?

5.5 Comment on treatment of quality control samples when scheduled for exposure.

5.6 Identify specific deficiencies; recommend improvements.

6.0 SAMPLE PRESERVATION

6.1 Breath collections: Is Drierite or some other drying agent placed in bottom of culture tubes?

6.2 Water collections: Do water collection bottles contain sodium thiosulfate?

6.3 Other collections:?

6.4 Comment on specific deficiencies of preservation methods and/or recommend measure which would benefit these procedures.

7.0 SAMPLE STORAGE

7.1 Describe manner in which samples are stored in Workroom.

Air, Breath:

Water:

Other:

7.2 Comment on specific deficiencies with regard to sample storage and/or recommend measures which would benefit these procedures. Comment on adherence to Sample Storage SOP.

8.0 CHAIN-OF-CUSTODY FORMS

8.1 Review the completed chain-of-custody/sampling protocol forms and check for the following:

(a) legibility -

(b) completeness -

(c) QA entries -

(d) consistency (e.g., sample volume values on front and back of sheet) -

8.2 Comment on specific deficiencies in implementation of chain-of-custody procedures and/or recommend measures which would benefit this phase of the study. Comment on adherence to chain-of-custody SOP.

9.0 SAMPLING SCHEDULE

9.1 Are the sampling schedules being followed?

9.2 What extenuating circumstances have caused a variance with the sampling schedule?

9.3 When necessary, are the modifications in the sampling schedule explained and documented?

9.4 Comment on specific deficiencies of the sampling schedule and/or recommend measures which would benefit this scheme.

10.0 CALIBRATION OF COLLECTION DEVICES

10.1 Air Collections:
When/where are the personnel air pumps calibrated?

How many individual calibration determinations are averaged to give rate reported on Sampling Protocol form?

10.2 Breath Collections:
When were Nu-Tech pumps last calibrated?

10.3 Other Collections:

10.4 Comment on specific deficiencies of calibration procedures and/or recommend measures which would benefit this operation. Comment on adherence to Calibration SOPs.

11.0 WORK LOAD

11.1 Is work load being distributed fairly among sampling teams?

11.2 Comment on whether work load tends to be (a) too excessive; potential of compromising QC and sampling methodology, (b) as projected; reasonable balance between sampling activity and free time, and (c) too slow; evidence of idle time and unexpected efficiencies in sampling effort.

12.0 CASH INCENTIVE PROCEDURES

12.1 Are sufficient funds on hand for the projected amount of cash incentive needed?

12.2 Where are funds maintained?

12.3 What is the mechanism for withdrawal of incentive cash? Be specific.

12.4 Is there a person responsible for the maintenance of incentive funds and for providing other sample collection teams with necessary cash incentives? Identify this person.

12.5 Are the receipt copies for funds dispersed being maintained in an orderly fashion, and are periodic balance checks being made against these receipts?

12.6 Comments on any part of the cash incentive procedure which is deficient, and/or recommend measures which should benefit the overall incentive protocol:

13.0 COLLECTION AND MANAGEMENT OF SURVEY INSTRUMENTS

13.1 List documents picked up by sampling team during initial visit to participant. Describe manner in which these documents are stored in the Workroom, the mode of shipment to RTI, and their fate on arrival at RTI.

13.2 List documents generated as a result of sampling team activity. Describe manner in which these documents are stored in the Workroom, the mode of shipment to RTI, and their fate on arrival at RTI.

13.3 Comment on specific deficiencies of the survey instrument procedure, and/or recommend measures to benefit same.

14.0 SAMPLE SHIPMENT TO RTI

14.1 Describe the manner in which samples are packaged (use of bubble-wrap or other packaging materials or containers).

Air, Breath:

Water:

Other:

14.2 Describe means for chilling/freezing samplings during transit.

Air Breath:

Water:

Other:

14.3 Describe shipping containers.

Air, Breath:

Water:

Other:

14.4 How are samples being shipped back to RTI, and what is the expected time of transit? Mention carrier.

Air, Breath:

Water:

Other:

14.5 Comment on specific deficiencies of the sample shipment procedures and/or recommend measures which would benefit the overall sample shipment protocol. Comment on adherence to Sample Shipment SOP.

15.0 MISCELLANEOUS

15.1 Indicate any unexpected problems or observations noted in any phase of the study.

15.2 Indicate overall assessment of sample collection activity.

15.3 Does field staff have names of local authorities in the event of an accident or emergency (e.g., local health official)?

RTI/ACS-SOP-860-001
May 1984
Revision 1

Standard Operating Procedure for Preparing Quality Control Samples on Tenax Cartridges

Author:

Nora P. Castillo
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Approved:

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Laboratory Manager, RTI/ACS Date

RW Handy 6/4/84
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E Pellizzari 6/4/84
Vice-President, RTI/ACS Date

Standard Operating Procedure for Preparing Quality Control Samples on Tenax Cartridges

RTI/ACS-SOP-860-001

1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure is three-fold: (1) it describes the steps to be followed to select and label the quality control samples; (2) it describes the loading of Tenax control cartridges with volatile organic compounds via a flash evaporation system (FES); and (3) it describes the methodology for loading volatile organic compounds onto Tenax control cartridges from a Permeation System (PS).

There are two types of quality samples: (1) spiked cartridges called "controls" and (2) unspiked cartridges called "blanks". The spiked cartridges (controls) allow determination of recoveries of the target compounds after sampling. The unspiked cartridges (blanks) are used to determine the background of the Tenax cartridges and contamination associated with sample transport and sample.

A brief description of the two systems follows.

1.1 Flash Evaporation System

The main body of the flash evaporation system consits of a glass tube with an injection port. The tube is maintained at 250°C with heating tape connected to a variable transformer.

A regulated helium stream transports any solution injected into the port to a sorbent cartridge connected downstream (see Figure 1).

1.2 Permeation System

A permeation system consists of three main parts: (1) a temperature-controlled chamber containing permeation tubes; (2) a mixing chamber; and (3) two cartridge holders enclosed in an insulated jacket. A stream of nitrogen circulates through the system. The amount(s) of compound(s) transported downstream remain(s) constant once the walls of the system become equilibrated with the compounds to be loaded (see Figure 2).

This Standard Procedure is used to prepare quality control samples for breath and air (QC sets).

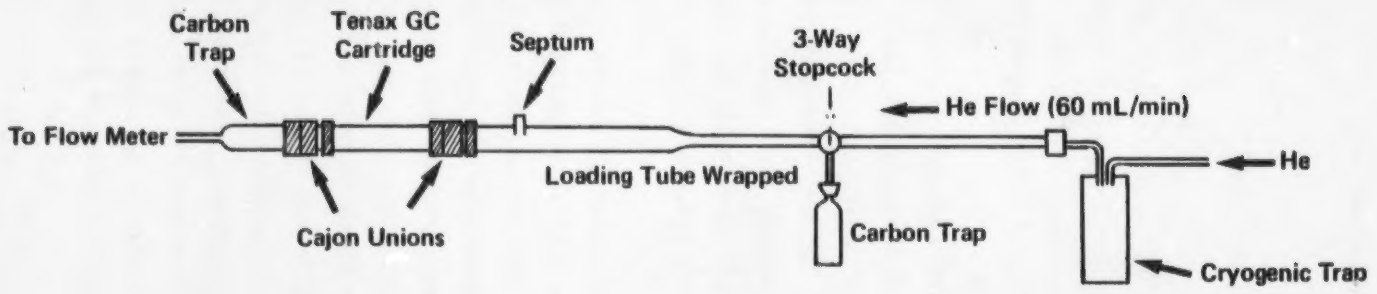
Carbon
Trap
Tenax GC
Cartridge
Septum
3-Way
Stopcock
He Flow (60 mL/min)
To Flow Meter
He
Loading Tube Wrapped
Cajon Unions
Carbon Trap
Cryogenic Trap

Figure 1. Flash evaporation system.

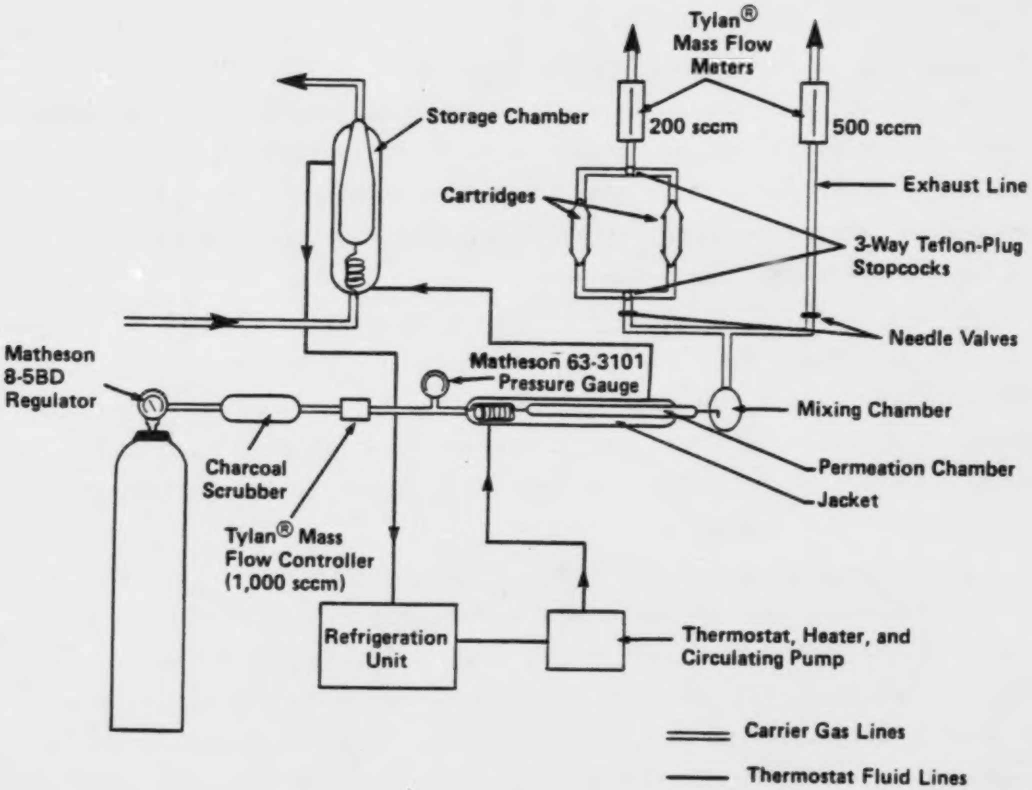
Tylan® Mass Flow Meters
Storage Chamber
200 sccm
500 sccm
Cartridges
Exhaust Line
3-Way Teflon-Plug Stopcocks
Needle Valves
Matheson 8-5BD Regulator
Matheson 63-3101 Pressure Gauge
Mixing Chamber
Charcoal Scrubber
Permeation Chamber
Jacket
Tylan® Mass Flow Controller (1,000 sccm)
Refrigeration Unit
Thermostat, Heater, and Circulating Pump
Carrier Gas Lines
Thermostat Fluid Lines

Figure 2. Permeation system for generating air/vapor mixtures.

*QC sets differ according to the matrix and the requirements of the study. An air QC set consists of one blank and two spiked cartridges or one blank and one spiked cartridge. A breath QC set consists of one blank, one spirometer blank and two spiked cartridges or only one spirometer blank and one spiked cartridge which contains all the target compounds.

2.0 SUMMARY OF THE METHOD

2.1 Selection of the Quality Control Samples

The selection of the quality control samples involves the following steps: (1) selection of the target compounds to be loaded; (2) determination of the number of control sets to prepare by matrix; (3) labeling the control sets to be prepared; and (4) preparation and storage of the unspiked cartridges (blanks) prepared.

2.2 Loading Control Cartridges via a Flash Evaporation System

This method includes: (1) the preparation of a methanol solution containing the target compounds; (2) the steps followed in the actual loading of a Tenax cartridge; and (3) the packing and storing of the controls loaded. In addition, this method provides quality assurance guidelines for the loading procedure.

2.3 Loading Control Cartridges From a Permeation System

The following routine shall be followed when Tenax cartridges, to be used as quality control, are loaded with target compounds via a permeation system: (1) selecting the permeation tubes; (2) determining the loading conditions to be used; (3) equilibrating the system; (4) loading the cartridges; (5) calculating the amounts of compounds loaded; and (6) packing and storing the cartridges. In addition, as in Section 2.2, this method provides quality assurance guidelines for the loading procedure.

3.0 IDENTIFYING THE QUALITY CONTROL SAMPLES

3.1 Selection of the Target Compounds to be Loaded

3.1.1 In a bound RTI notebook, assigned to the project, list all the compounds to be loaded.

3.1.2 Reject permeation tubes whose permeation rates are below 100 ng/min or above 600 ng/min.

CAUTION

Do not select any permeation tube whose permeation rate is unstable. A permeation rate is considered stable when the mean permeation rate has a coefficient of variation (CV) of less than 10%. The mean permeation rate is calculated using the last five individual permeation rates.

3.1.3 In the notebook, prepare a table including: (1) the numbers of the selected permeation tubes; (2) the compound names; and (3) the corresponding mean permeation rates.

Tube #	Compound	Permeation Rate Mean ± S.D. (CV)

3.1.4 Prepare a second table containing compounds to be loaded using the flash evaporation system, including compound names and their respective densities.

NOTE: The procedure for loading the cartridges is outlined in Sections 4.0 and 5.0.

3.2 Determination of the Number of Quality Control Sets to be Prepared

3.2.1 Obtain a copy of the Field Sampling Schedule from the Quality Assurance Officer.

3.2.2 Inquire as to the number of participants to be sampled from the person in charge of the field sampling trip.

3.2.3 Inquire as to the number of batches of Tenax prepared for the trip and the matrix to which each batch is assigned, from the person in charge of Tenax preparation.

NOTE: A batch of Tenax consists of all Tenax subjected to the cleanup process at the same time (see SOP-___-XXX for details). A batch of Tenax normally comprises a minimum of 80 cartridges and a maximum of 150 cartridges.

3.2.4 From the Field Sampling Schedule, determine the number of participants that will be sampled using each Tenax batch.

3.2.4.1 Include the number of duplicate cartridges for each participant.

3.2.4.2 Include the number of field control sets to be exposed for a given participant (if any).

CAUTION

*Note that a control set includes a blank and one or two spiked cartridges.

*3.2.4.3 Include laboratory control sets (3% of the field sample cartridges).

*3.2.4.4 Include two extra QC sets per batch to be analyzed by GC/FID.

CAUTION

Increase the number of cartridges required per batch by at least 5% in the event of breakage and for use as extra laboratory blanks.

NOTE: The Quality Assurance samples are analyzed by a Quality Assurance Laboratory.

CAUTION

Make allowance for 10% of the batch to be used for QC cartridges or a minimum of three QC sets for the Quality Assurance Laboratory.

3.2.6 Write a memo to the person in charge of Tenax cartridge preparation requesting the total number of cartridges required. Indicate the number of cartridges per batch and the date they need to be ready.

NOTE: The control sets need to be prepared approximately two days in advance of the sampling trip.

CAUTION

Ensure that the Tenax background has been checked and is acceptable before loading the cartridges. Ensure that the Tenax cartridges have been selected from different desorption dates.

3.3 Labelling the Cartridges

3.3.1 Request the preprinted labels from the person in charge of the field trip.

NOTE: The labels are preprinted with the project identifier and the respondent number. For all control sets, the respondent number will be a unique unit number.

*3.3.2 Complete the sample code on the labels by adding the following information: matrix - sample type - the number corresponding to the order it was prepared and must be exposed.

3.3.3 Prepare the labels in triplicate.

3.3.4 Transfer one label to the protocol sheet as follows:

FIELD SAMPLING PROTOCOL SHEET - TEAM STUDY

DATE: Date prepared

SAMPLE CODE: TEAM 00226-1
AV-FB-01

AIR SAMPLING

3.3.5 Transfer the second label to the COC record on the back of the PS sheet and indicate the Tenax batch number and sample type.

CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute, Analytical Sciences Division
Chemistry and Life Sciences Group, Research Triangle Park, NC 27709

SAMPLE CODE TEAM 00226-1 AV-FB-01	**TENAX BATCH:** Batch #	**VOLUME COLLECTED:**
SAMPLE TYPE: TENAX	**TEMPERATURE (°F)**	**VOLUME ANALYZED:**

Table 1

Matrix

Personal Air = AV

Fixed Site Air = XV

Breath = BR

Sample Type

Field Blank = FB

Field Control = FC

Spirometer Blank = SB

Laboratory Blank = LB

Laboratory Control = LC

3.3.6 Attach the third label to the Kimax tube containing the appropriate Tenax cartridge.

CAUTION

Ensure that the cartridge belongs to the Tenax batch assigned for that sample. The batch number is indicated on the screw-cap of the Kimax culture tubes.

3.3.7 Secure the label with transparent tape.

3.3.8 Separate the cartridges designated as blanks from the controls.

3.3.9 Store the cartridges designated as controls in a sealed paint can in the clean room until loading.

3.4 Storage of Blank Cartridges

3.4.1 Separate the field blanks from laboratory blanks.

3.4.2 Secure the field blanks inside the Kimax tube with a glass wool plug to avoid breakage during transport.

3.4.3 Store the field and laboratory blanks in separate sealed paint cans in the freezer until they are ready to be sent to the field or to the Mass Spectrometry laboratory for analysis.

4.0 PROCEDURE FOR LOADING CONTROLS USING THE FLASH EVAPORATION SYSTEM

4.1 Preparation of the Solution

4.1.1 Apparatus and Materials

4.1.1.1 Capillary pipet, 50 μL
4.1.1.2 Capillary pipet, 100 μL
4.1.1.3 Volumetric flask, 5 mL
4.1.1.4 Volumetric flask, 10 mL
4.1.1.5 Disposable gloves
4.1.1.6 Small spatula
4.1.1.7 Microbalance
4.1.1.8 Pasteur pipets
4.1.1.9 Weighing boat, small, aluminum
4.1.1.10 Vials, screw cap (open top), 4 mL
4.1.1.11 Septum, silicone rubber, Teflon-faced

4.1.2 Reagents

4.1.2.1 Neat compounds (purity >97%)

4.1.2.2 Methyl alcohol, B&J distilled in glass

4.1.3 Procedure

4.1.3.1 In a bound RTI notebook, make a list of the compounds to be loaded and their respective densities.

*4.1.3.2 Calculate the amount required to prepare a stock solution of ~8-10 µg/µL per analyte in a 10 mL volumetric flask, using 50 or 100 microliter capillary pipets.

4.1.3.3 Prepare a table that includes:

Compound Name	Density (mg/µL)	µL Loaded	mg/5 mL	µg/µL

4.1.3.4 Ensure that all the compounds are on hand before beginning preparation of the solution.

WARNING

Use gloves and a hood when handling these chemicals as they may be dangerous to your health.

4.1.3.5 If the mixture includes any solid compound, use a microbalance. Weigh the compound in an aluminum weigh boat and transfer it quantitatively to the volumetric flask.

WARNING

Do not spend too much time weighing the compound(s).

4.1.3.6 Add ~0.5 mL of methyl alcohol to the volumetric flask.

4.1.3.7 Aliquot all liquids in a hood, one at the time, using capillary pipets.

4.1.3.8 Rinse the walls of the flask with small quantities of methyl alcohol using a Pasteur pipet to avoid volatility losses.

4.1.3.9 After the last compound has been added stopper the volumetric flask and invert it five times to mix. Dilute to volume with methyl alcohol.

4.1.3.10 Label the flask "Control Mxt. Stock Solution". Include the date of preparation and your initials.

4.1.3.11 Mix well. Transfer 100 µL of the stock solution to a 10 mL volumetric flask.

4.1.3.12 Dilute to volume. Mix well and transfer to 3 screw-cap vials with silicone septa.

4.1.3.13 Label them as Control Mxt. Solution 1, 2 and 3. Include the date of preparation, your initials and the page of the notebook where all the information was recorded.

4.1.3.14 Transfer the stock solution to a screw-cap vial and record all pertinent information.

4.1.3.15 Store all standard solutions at 4°C protected from the light.

4.1.3.16 Open a new vial of the diluted solution every 6 weeks. Discard the previous solution.

4.1.3.17 Use the concentrated stock solution to prepare other dilutions or CPMX/RMR solutions for up to 6 months.

4.2 Use of the Flash Evaporation System

4.2.1 Materials and Apparatus

4.2.1.1 Flash evaporation system (see Figure 1)
4.2.1.2 Helium, 2500 psi cylinder (certified 99.995%) with regulator
4.2.1.3 Stopwatch
4.2.1.4 Kimwipes®
4.2.1.5 Hamilton micro-syringe, 5 µL
4.2.1.6 Septum, half-hole type, Supelco LB-1, or equivalent
4.2.1.7 Tweezers
4.2.1.8 Insulated gloves
4.2.1.9 Methyl alcohol, B&J distilled in glass
4.2.1.10 Silicone rubber septum, Teflon-faced

4.2.2 Loading of the Control Cartridges

Ensure that cartridges have been checked for satisfactory background before beginning loading.

4.2.2.1 Turn on helium gas.

4.2.2.2 After 2 or 3 minutes, fill the cryogenic trap with liquid nitrogen.

WARNING

Introducing the liquid nitrogen before the helium is flowing will cause any water that may be present in the cryogenic trap to freeze and prevent circulation of the helium through the system.

4.2.2.3 Rotate the stopcock to direct gas flow through the loading chamber.

4.2.2.4 Adjust the flow with the needle valve to approximately 60 mL/min, using the stopwatch and bubble meter.

4.2.2.5 Calculate flow in mL/min according to the equation:

$$\text{Flow (mL/min)} = \frac{\text{volume (mL)}}{\text{time (sec)}} \times \frac{60\ \text{sec/min}}{1\ \text{min}}$$

4.2.2.6 Repeat the measurement twice and record the average.

4.2.2.7 Rinse the syringe several times with clean methyl alcohol.

CAUTION

Allow the control solution to reach room temperature and then sonicate it for five minutes before using.

4.2.2.8 Attach the Tenax cartridge line with the bottom part toward the loading chamber (see Figure 3).

CAUTION

To avoid contamination, always handle the Tenax cartridge with Kimwipes® or clean nylon gloves.

4.2.2.9 Inject the appropriate volume of the solution to be loaded (normally 2 μL) through the septum, holding the needle in place for 15 sec.

4.2.2.10 Simultaneous with the injection, start the stopwatch.

*4.2.2.11 Pass helium through the cartridge at ~60 mL/min for 10 min (~600 mL total).

NOTE: The breakthrough volume for methyl alcohol is 600 mL.

4.2.2.12 Turn the stopcock to the exhaust position to stop the flow through the system.

4.2.2.13 Return the cartridge to the culture tube. Seal the tube.

4.2.2.14 Label the culture tube with the preprinted label assigned to the sample.

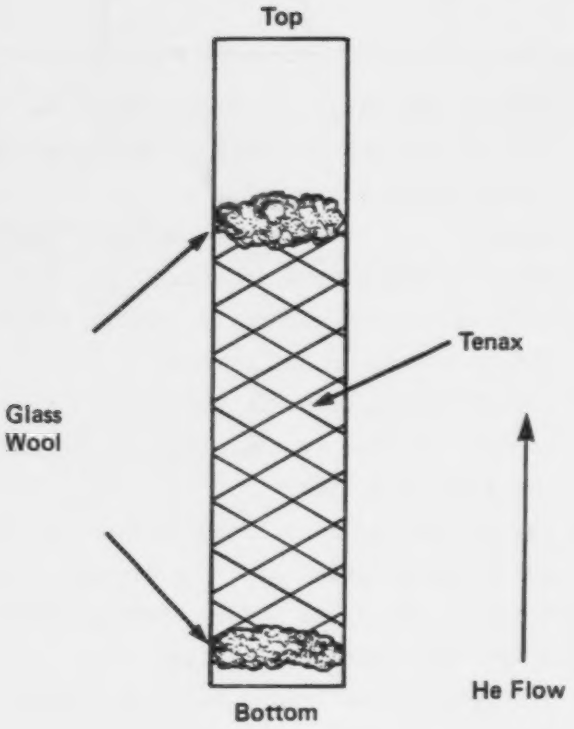
Top
Tenax
Glass
Wool
He Flow
Bottom

Figure 3. Tenax cartridge.

4.2.2.15 On the SP/COC sheet already prepared for the control, include the following in preparation:

RELIN-QUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	(Your initials)		Date of Loading	2 μL Control Mxt. from FES

4.2.2.16 Analyze the first cartridge loaded by GC/FID according to the conditions specified in the QA section of SOP-320-001 (Preparation of Tenax).

4.2.2.17 Refer to the Quality Assurance Section of this SOP (Section 6.0) for additional details.

4.2.2.18 To load another cartridge, rotate the stopcock to direct the flow through the system.

4.2.2.19 Follow instructions in steps 4.2.2.8 to 4.2.2.15.

4.2.2.20 To turn off the system close the helium tank. Maintain the system at 250°C.

4.2.2.21 Change the Teflon-silicone septum in the solution vial.

4.2.2.22 Store the solution at 4°C protected from the light.

4.2.2.23 Separate the field and laboratory control cartridges.

*4.2.2.24 Store the control cartridges in the freezer until they are ready to be loaded from the permeation system or ready to be sent to the field.

5.0 PROCEDURE FOR LOADING CONTROLS FROM THE PERMEATION SYSTEM

5.1 Materials and Equipment

5.1.1 Permeation system (see Figure 1)
5.1.2 Nitrogen gas (certified 99.995%)
5.1.3 Nylon gloves
5.1.4 Long glass hook (for retrieving permeation tubes)

5.1.5 Permeation tubes

5.1.6 Kimwipes®

5.1.7 Stopwatch

5.2 Transfer of the Permeation Tubes to the Loading Chamber

5.2.1 Turn off the nitrogen flow in the storage chambers (see Figure 2).

5.2.2 Locate the chambers in which the selected permeation tubes are stored.

NOTE: Permeation tubes are stored in numerical order in the chambers. The range of numbers contained therein is listed outside each chamber.

WARNING

Open one storage chamber at a time. They contain toxic materials that are dangerous to your health.

5.2.3 Open the loading chamber as follows:

5.2.3.1 Lower the jack which supports the chamber.

5.2.3.2 Remove the clamp securing the two chamber sections.

5.2.3.3 Carefully open the loading chamber by moving the downstream section to the rear.

5.2.3.4 Remove the "O" ring.

WARNING

Failure to wear nylon gloves when handling permeation tubes may result in skin irritation and/or contamination of the permeation tubes.

5.2.4 With a long glass hook, remove the selected permeation tubes from the storage chamber and transfer immediately to the loading chamber.

WARNING

Perform this operation as quickly as possible. The tubes contain toxic materials, some of which are suspected cancer suspect agents.

5.2.5 Reposition the "O" ring and reconnect the two sections of the loading chamber.

5.2.6 Attach the clamp.

5.2.7 Close and secure the storage chambers. Initiate nitrogen flow by opening the needle valve connected to the "house" nitrogen line.

CAUTION

Ensure that the rotameter indicates sufficient flow to distribute approximately 500 mL/min through each storage chamber.

5.3 Operation of the Permeation System

5.3.1 Turn on the read-out box.

NOTE: The read-out box displays the measurements of the mass flow meter and the mass flow controllers. The labels on the box indicate the proper channels.

CAUTION

Ensure that the pressure regulator on the nitrogen tank is fully open counterwise (i.e., no pressure).

5.3.2 Turn on the nitrogen gas.

CAUTION

Ensure that the read-out box power is on before any nitrogen circulates through the system, otherwise the controller will be fully open and the excessive nitrogen flow will damage the system.

5.3.3 Carefully, increase the nitrogen pressure to the permeation system by rotating the pressure regulator T-handle clockwise, observing the gauge in the system at all times.

CAUTION

Do not allow the pressure on this gauge to exceed 10 inches of water.

5.3.4 Set the outlet pressure at the nitrogen cylinder to 20 psi.

5.4 Determining the Approximate Flow Rates for Loading

5.4.1 Calculations

5.4.1.1 For any compound, calculate the amount loaded onto a cartridge using the following formula:

$$G = Pt \frac{F_1}{F_1 + F_2}$$

where:

G = amount loaded (ng)
P = permeation rate (ng/min)
t = time (min)
F_1 = flow rate through the cartridge (mL/min)
F_2 = exhaust flow rate (mL/min)

5.4.1.2 Fix the time (t), amount to be loaded (G), and flow rate through the cartridge (F_1) and calculate exhaust flow rate (F_2).

5.4.1.3 Use the following example:

Objective: to load 100 ng of chloroform whose P = 130 ng/min

Fix: t = 4 min

F_1 = 85 mL/min

F_2 = ?

Calculate:

$$G = (P)\ (t)\ \frac{F_1}{F_1 + F_2}$$

$$F_2 = \frac{(P)\ (t)\ (F1)}{G} - F_1$$

$$F_2 = \frac{(130\ \text{ng/min})\ (4\ \text{min})\ (85\ \text{mL/min})}{100\ \text{ng}} - 85\ \text{mL/min}$$

$$F_2 = 357\ \text{mL/min}$$

NOTE: For more examples see SOP-630-001 (loading of RMR cartridges).

5.5 Setting the Flow Rates for the Loading

5.5.1 Enter the determinated flow rates and time in the notebook.

CAUTION

Do not load a cartridge using a cartridge flow below 50 mL/min or above 150 mL/min. Do not operate the system with a total flow below 250 mL/min or above 600 mL/min. Do not load for less than two minutes. These restrictions increase the accuracy and precision of the loading process.

5.5.2 Rotate the two stopcocks to direct the nitrogen flow to the side where the Tenax cartridge will be loaded (see Figure 1).

NOTE: When a cartridge is not being loaded, a dummy cartridge is placed in the loading position.

5.5.3 Turn the knob on the read-out box to the total flow rate channel.

5.5.4 Adjust the total flow rate with the appropriate controller adjustment knob.

5.5.5 Stabilize the flow rates for two minutes.

5.5.6 Turn the knob to the cartridge flow rate channel. Adjust the nitrogen flow rate through the cartridge with the proper needle valve.

5.5.7 Turn the knob to the exhaust flow rate channel.

5.5.8 Read the exhaust flow rate.

NOTE: The exhaust flow rate is equal to the total flow rate minus the flow rate through the cartridge.

CAUTION

If the total flow rate does not equal the sum of the exhaust flow rate and the cartridge flow rate, a gas leak should be suspected. Ensure that the "O" ring is properly sealed and the clamp is secure.

5.6 Equilibration of the System

5.6.1 Allow the system to equilibrate for 90 minutes before starting the loading.

5.6.2 Reread the flow rates on the various channels before starting the loading.

5.7 Loading of the Tenax Cartridges

To ensure that the system is working properly, load a control cartridge, following the loading instructions in this SOP and analyze it as indicated in the Quality Assurance Section.

5.7.1 Divert the nitrogen flow to the side that will not be used for loading cartridges (see Figure 1) by turning the two stopcocks clockwise, simultaneously.

5.7.2 Loosen the Cajon® fittings and insert the Tenax cartridge with the bottom part down (see Figure 3).

WARNING

To avoid contamination, use Kimwipes® when handling the cartridge.

5.7.3 Tighten the bottom fitting first, then the upper one.

5.7.4 Rotate the two stopcocks clockwise, simultaneously, to direct the gas flow through the cartridge.

5.7.5 Concurrently, start the stopwatch.

5.7.6 Turn the knob on the read-out box to the cartridge flow rate channel. Record this flow rate in the notebook.

5.7.7 Turn the knob to the channel displaying the exhaust flow rate. Record this flow rate in the notebook.

5.7.8 Calculate the time needed to load the amounts desired as follows:

$$t = \frac{G}{P} \times \frac{F_1 + F_2}{F_1}$$

(See Section 3.2.1 for definitions). Base this calculation on the compound whose permeation tube has the highest or lowest permeation rate.

5.7.9 At the calculated time rotate the two stopcocks to direct gas flow away from the cartridge being loaded.

5.7.10 Handling the cartridge with a Kimwipe®, loosen the Cajon fittings bottom side first.

5.7.11 Remove the cartridge and return it to its culture tube. Seal the tube.

5.7.12 In the SP/COC sheet already prepared for the control include the following information:

RELIN-QUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	(Your initials)		Date of Loading	Control Mxt. from Perm. System

5.7.13 Analyze the first cartridge by GC/FID as indicated before. See QA section for more details.

5.7.14 To load another control cartridge follow instructions in Steps 5.7.1 to 5.7.13.

5.8 Turning Off the Permeation System

5.8.1 Open the pressure regulator slowly (counterwise) until fully open.

5.8.2 When the pressure gauge indicates no pressure, open the loading chamber as follows: (1) lower the jack; (2) remove the clamp; (3) carefully move the upper part of the system back; (4) use jack to support the system; and (5) remove the "O" ring.

5.8.3 Turn off the nitrogen flow in the storage chambers.

WARNING

Failure to wear nylon gloves when handling permeation tubes may cause skin irritation and/or contamination of the permeation tubes.

5.8.4 With a long glass hook, remove one tube at a time, from the loading chamber and transfer immediately to the proper storage chamber.

5.8.5 After the last tube has been transferred reclamp the storage chambers.

5.8.6 With the needle valve that is connected to the "house" nitrogen line, turn the nitrogen gas on.

5.8.7 Reposition the "O" ring on the loading chamber and move the upper part of the system back to the normal position.

5.8.8 Attach the clamp.

5.8.9 Turn off the read-out box.

5.9 Storage of the Control Cartridges

5.9.1 Separate the field controls (FC) from laboratory controls (LC).

5.9.2 Store the LC cartridges in a sealed paint can in the freezer assigned to that purpose.

5.9.3 Secure the FC cartridges inside the Kimax® tube with a glass wool plug to avoid breakage during transport.

5.9.4 Store the FC cartridges in a sealed paint can in the freezer until they are ready to be sent to the field.

5.10 Calculation of the Amounts Loaded

5.10.1 For each compound, calculate the amount loaded onto a cartridge using the general formula:

$$G = Pt \frac{F_1}{F_1 + F_2}$$

(See Section 5.4 for definitions.)

5.10.2 In the notebook, prepare a table that includes: (1) the control codes; (2) the date of preparation; (3) the compounds loaded; and (4) the amounts (in ng/cartridge) loaded for each compound.

6.0 QUALITY ASSURANCE FOR THE LOADING OF CONTROLS

6.1 Controls from Flash Evaporation System

6.1.1 Analyze the first cartridge loaded using a Varian 3700 gas chromatograph equipped with a thermal desorption injection unit and a flame ionization detector and interfaced to a Varian CDS 111 integrator.

6.1.2 Analyze the cartridge using the following instrument conditions:

6.1.2.1 Column: Fused silica capillary column (60 m x 0.25 μm); Liq. phase DB1; or an equivalent column

6.1.2.2 Detector temp.: 300°C

6.1.2.3 Injector temp.: 280°C

6.1.2.4 Desorption unit temp.: 260°C

6.1.2.5 Temp. Program: 30°C (2 min), 4 °C/min to 220° (10 min)

6.1.2.6 Attenuation: 128×10^{-12}

6.1.2.7 Recorder chart speed: 1 cm/min

6.1.2.8 He carrier flow: 1.2 mL/min

6.1.2.9 H_2 flow to FID: 30 mL/min

6.1.2.10 Air flow to FID: 300 mL/min

6.1.2.11 Make-up gas: ~30 mL/min

6.1.2.12 For each compound, calculate the instrumental response factor using the following formula:

$$\text{Response Factor} = \frac{\text{Area Counts}}{\text{ng loaded}}$$

6.1.4 Compare the calculated response factor with historical response factor generated under identical conditions.

NOTE: The historical data is stored as the mean response factor and a standard deviation for each compound. A response factor is considered out-of-control when it deviates from the mean response factor by more than three standard deviations (3σ). This data is stored in a bound RTI notebook, assigned only for the instrument used.

6.1.5 If a mean response factor does not exist, create one after loading three cartridges and calculate the mean response factors.

6.1.6 If any major alterations are made to the GC (i.e., replacement of the column; replacement of the thermal desorption/injection unit; cleaning of the detector), create a new mean response factor.

6.1.7 If the analysis of the first cartridge indicates that less than two compounds are out of control, begin loading the cartridges.

6.1.8 If two or more are out-of-control, load a second cartridge and analyze it.

6.1.9 If the second cartridge is in control proceed with the loading.

6.1.10 If it is out-of-control, load and analyze a column performance evaluation (CPMX) cartridge with a column performance mixture solution following steps 4.2.2.7 to 4.2.2.13.

NOTE: The analysis of a CPMX cartridge provides a check on the instrument conditions.

6.1.11 If the CPMX cartridge is in control, prepare a new solution as indicated in Section 4.1 and repeat the analysis.

6.1.12 If the CPMX cartridge is out-of-control, troubleshoot the instrument before proceeding with the loading.

6.2 Controls from Permeation System

6.2.1 Follow the same general instructions as for control from FES (steps 6.1.1 to 6.1.11 in Section 6.1).

6.2.2 If the CPMX cartridge is in control check the permeation system for leaks.

CAUTION

Positive results from a CPM analysis indicates a failure in the permeation system. Be sure the problem is solved before loading controls.

7.0 RELINQUISHING THE CONTROL SETS

7.1 Take the sealed paint cans out of the freezer.

7.2 In a clean room, open the cans and separate the cartridges in sets by matrix and codes.

7.3 Label the top of each cartridge with the sequence number on the respective code.

Example: 00226-1-AV-FB-01

Label the top with a circle 01.

7.4 Organize them in sealed paint cans by sets and matrix.

7.5 Do not store more than 17 cartridges in a paint can.

7.6 Seal the cans and label them on the top indicating the content of the can.

Example: Field Control Sets #1, #2, #3 for Breath.

7.7 Sign the SP/COC sheets as relinquished.

7.8 Deliver the cans and the SP/COC sheets to the person in charge of the packing and shipment of Tenax cartridges.

RTI/ACS-SOP-861-002 (Air)
SOP-862-002 (Water)
SOP-867-002 (Breath)
December 1983
Revision 0

Standard Operating Procedure for the Shipment of Quality Control Samples to the Field Sampling Site

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Standard Operating Procedure for the Shipment of Quality Control Samples to the Field Sampling Site

RTI/ACS-SOP-861-002 (Air)
SOP-862-002 (Water)
SOP-867-002 (Breath)

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure describes the method for shipping Quality Control (QC) samples of all matrices to the field sampling site. It describes: (1) checking the samples prepared against Sampling Protocol/ Chain-of-Custody (SP/COC) sheets; (2) documenting this check plus receipt and transmittal of the samples on COC records; and (3) proper packing of samples for transport to the field.

2.0 SUMMARY OF THE METHOD

All QC samples undergo a routine procedure for shipment to the field. Samples are matched to the COC sheets and then packed for shipment. The QC Tenax samples plus SP/COC sheets are packed in a footlocker with other cartridges of the same batch(es) and carried to the RTI Inventory Control Clerk or Shipping and Supply Specialist for shipment by air to the field sampling site. QC water samples are packed in small coolers and alternatively transported to the field in the same van(s) used to convey sampling equipment to the field site.

3.0 PROCEDURE

3.1 Tenax Air and Breath QC Samples

3.1.1 Field Blanks and Field Control Sites

3.1.1.1 Locate these QC sets in the freezer designated for Tenax cartridge storage.

NOTE: These sets are in separate cans by matrix (personal air, fixed-site, and breath), or combined in the case of a small study. The can will be labelled "QC Field Sets".

3.1.1.2 Secure the SP/COC sheets from the staff member who prepared the QC sets.

3.1.1.3 Carry the can(s) containing the QC field sets to the "clean room" and open them.

3.1.1.4 Match a SP/COC sheet to each sample.

3.1.1.5 Notify the staff member who prepared the QC sets of any discrepancies.

3.1.1.6 Enter the following information on each COC record.

RELINQUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	(Your initials)		The date of the check	QA; to the field
(Your initials)				

3.1.1.7 Check to ensure that all of the culture tubes are packed with glass wool between the top of the cartridge and the screw cap so that the Tenax cartridge is immobile when shaken.

3.1.1.8 If any culture tubes lack glass wool, insert (using forceps) an unsilanized glass wool plug in each tube.

CAUTION

Glass wool protruding from the sides of the cap will reduce the effectiveness of the seal and may result in contamination of the Tenax sorbent bed.

3.1.1.9 Place the sealed culture tubes in the can.

3.1.1.10 Tightly pack each can with cotton, ensuring that each culture tube is surrounded. Place sufficient cotton on top of the tubes so that the culture tubes do not move when the can is sealed and shaken.

3.1.1.11 Seal each can.

3.1.1.12 Place the can(s) in a footlocker with cans containing packed cartridges of the same batch(es) to be used in the field sampling operation.

3.1.1.13 Place a maximum of eight (8) cans of cartridges per footlocker.

3.1.1.14 Ensure that the cans are well-cushioned inside the footlocker (see Figure 1).

3.1.1.15 Place a foam pad on the floor and four sides of the footlocker.

3.1.1.16 Evenly space the eight (or less) cans of Tenax cartridges on top of the pad lining the bottom of the footlocker.

3.1.1.17 Place pieces of foam in between each can so as to isolate the cans.

3.1.1.18 Include the SP/COC sheets in the footlocker.

3.1.1.19 Place a piece of foam inside the top of the footlocker to secure the cans when the footlocker is closed.

3.1.1.20 Close and lock the footlocker.

3.1.1.21 Complete a Shipment Clearance Sheet (see Figure 2).

3.1.1.22 Notify the RTI Inventory Control Clerk or Shipping and Supply Specialist of intended shipment.

3.1.1.23 Carry the footlocker and Shipment Clearance Sheet to either person listed in 3.1.1.22 above for subsequent air shipment to the field sampling site.

SHIPMENT CLEARANCE SHEET

Charge Project: ______

Person Requesting Shipment: ______

Date: ______

**NOTE: Please include street address - incomplete addresses are a constant source of trouble. Also include Zip Code and telephone numbers, if available.

Expiration date: ______

Consignee: ______

Requested Date of Shipment: ______

Address: ______

Requested Mode of Shipment:

Federal Express, 24 hr ______

48 hr ______

Courier Pak ______

Signature Service ______

US Mail ______

Other ______

For the Use of: ______

Insurance Value ______

Address: ______

Invoice Value ______

Telephone number: (   )

Please circle answer that applies and fill in any blanks which apply.

Solid? yes/no Liquid? yes/no If yes, solution? ______ (Name of Solvent)

Carcinogen? yes/no Poison? yes/no Unstable? yes/no

Ship in Dry Ice? yes/no Explosive (or subject to violent decomposition)? yes/no

Radioactive? yes/no If yes, radioisotope? ______

Exact Description of Material: ______

Comments, Special Instructions and Precautions: ______

Figure 2. Shipment clearance sheet.

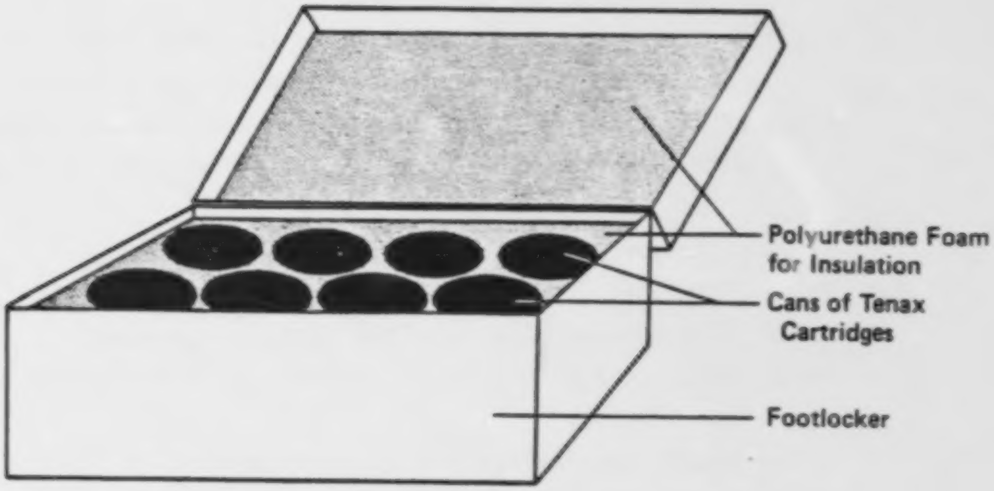
Polyurethane Foam for Insulation
Cans of Tenax Cartridges
Footlocker

Figure 1. Cans of Tenax cartridges cushioned inside a footlocker.

3.1.2 Deuterated Standards

3.1.2.1 Locate these cartridges in the freezer designated for Tenax cartridges only.

NOTE: The label "Deuterated Standards" is on the can containing the cartridges.

3.1.2.2 Secure the SP/COC sheets from the staff member who prepared the cartridges with deuterated standards.

3.1.2.3 Carry the can(s) to the "clean room" and open them.

3.1.2.4 Ensure that a SP/COC sheet exists for each cartridge, and that the batch numbers match.

3.1.2.5 Follow Steps 3.1.1.6-3.1.1.22 above.

3.2 Water QC Samples

3.2.1 Locate these QC sets in the TEAM refrigerator.

3.2.2 Obtain the SP/COC sheets from the staff member who prepared the QC sets.

3.2.3 Remove the samples from the refrigerator.

3.2.4 Match a SP/COC sheet with each sample.

3.2.5 Notify the staff member who prepared the QC sets of any discrepancies.

3.2.6 Enter the following information on each COC sheet.

RELINQUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	(Your initials)		The date of the check	QA; to the field
(Your initials)				

3.2.7 Ensure that the screw caps are secure.

3.2.8 Pack the water QC samples in a cooler.

NOTE: Any small cooler may be used. The Igloo® six-pack cooler will hold a maximum of 28 water samples.

3.2.8.1 Line the bottom and sides of the cooler with a layer of plastic bubble wrap.

3.2.8.2 Lay the water samples on their sides with bubble wrap surrounding each bottle so that no two bottles touch (see Figure 3).

3.2.8.3 Fill the cooler with layers of bottles and bubble wrap.

3.2.8.4 Fill all unfilled spaces in the cooler with bubble wrap so that the bottles are securely in place.

3.2.8.5 Put a layer of bubble wrap on top of the top row of samples.

3.2.8.6 Place two frozen blue-ice packs (or equivalent) on top of the bottles.

3.2.8.7 Close the cooler.

3.2.8.8 Put the COC sheets in an envelope with the cooler or the notebook which will be used to keep field sample COC sheets.

3.2.8.9 Keep the cooler in the walk-in refrigerator.

NOTE: Just prior to departure to the field, the sampling team will put the cooler in the van.

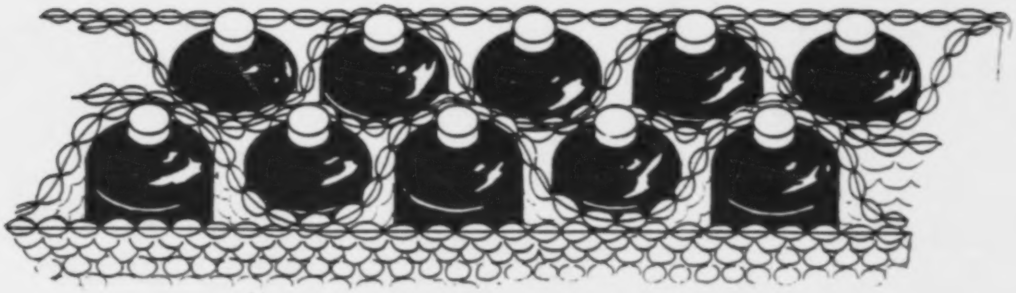

Figure 3. Cross-section of bottles insulated with plastic bubble wrap.

RTI/ACS-SOP-861-003 (Air)
SOP-862-003 (Water)
SOP-867-003 (Breath)
December 1983
Revision 0

Standard Operating Procedure for the Exposure of Quality Control Samples

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12/5/83
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Vice-President, RTI/ACS Date

Standard Operating Procedure for the Exposure of
Field Quality Control Samples
RTI/ACS-SOP-861-003 (Air)
SOP-862-003 (Water)
SOP-867-003 (Breath)

1.0 PRINCIPLE

During the course of a field sampling trip quality control samples for each matrix are exposed. Exposure of water, breath, personal air and fixed site air QC samples are discussed in this text.

2.0 MATERIALS

2.1 Quality control samples (blanks and controls)
2.2 Chain-of-Custody/Field Sampling Protocol Sheets
2.3 Drierite;, nonindicating; baked at 400°C for 2 hours
2.4 Ice cooler and ice packs
2.5 Spirometer apparatus

3.0 ASSIGNMENT OF QC SETS FOR EXPOSURE

QC sets are exposed at specific times during the sampling trip. The frequency of their exposure is reflected in the Sample Collection Schedules prepared by the project QA Officer. Each matrix has a collection schedule and the frequency of exposure of the QC sets are unique to that matrix.

3.1 Refer to the Sample Collection Schedule prepared by the project QA Officer (see Figure 1 for an example).

NOTE: Each matrix has a specific Sample Collection Schedule. In the event more than one sampling site is under study, each site shall have a complete set of Sample Collection Schedules specific for that site.

3.2 Determine which participant numbers are associated with the exposure of a Quality Control (QC) set.

Site (Season) — Matrix

Participant Number	Samples Collected Field	Duplicates	QA	Expose QC Set
21476-7	2			
21477-5	2			
21478-3	2			1
21479-1	2		2	
21480-9	2			
21481-7	2			
21482-5	2			
21483-3	2	2		1
21484-1	2			
21485-8	2			

Figure 1. Sample Collection Schedule (Example)

NOTE: For example, Figure 1 under the heading "Expose QC Set" indicates that a QC set is to be exposed in conjunction with the third participant listed. The first QC set listed should be designated set number one.

3.3 Obtain the Chain-of-Custody (COC)/Field Protocol Sheets (SP) for the assigned QC set (see Figures 2 and 3).

NOTE: Each FC and FB has its own COC sheet. They are prepared and prelabelled prior to the sampling trip. Each QC set is prelabelled.

3.4 Place a label bearing the sample code number of the designated participant under the "General Remarks" section of the SP sheet for the assigned QC set.

NOTE: In order to relate the exposure of a QC set to a specific set of samples, it is necessary to affix the participant's sample code number on the QC set's COC sheets.

4.0 EXPOSURE OF WATER QC SETS

QC sets for water volatiles consist of one FC and one FB. They are exposed during an appointment during which a water sample is collected from the participant.

4.1 Remove the assigned QC set from the storage refrigerator in the workroom and place into a portable cooler containing frozen ice packs.

4.1.1 In the event a FC or FB for the assigned QC set is damaged, lost, or destroyed before exposure, disregard the entire set. Assign the next numbered QC set in its place and repeat steps 3.3-3.5.

4.1.2 If a QC set is not exposed, indicate on the SP sheet of the affected sample its exact condition and on both COC and SP sheets for the set that the samples were "Not Exposed."

4.2 Carry the portable cooler to the sample collection site and use it to store all of the collected field samples as well as the QC set.

4.3 Expose the QC set during the second (early morning, second day) appointment.

NOTE: Generally three appointments are scheduled with a participant. Water samples are collected only during the second and final appointment. Exposure of the QC set may occur during either appointment; however, the second is preferable for several reasons. The second appointment is generally shorter, therefore, minimizing the QC set's exposure. Secondly, in the event the QC set is not exposed during the second appointment, it may be exposed during the final appointment.

FIELD SAMPLING PROTOCOL SHEET - TEAM STUDY

DATE: ____

SAMPLE CODE: INAIR 111-5 VO-FB-1

AIR SAMPLING

Pump Model: ____ Serial Number: ____

Time (Initial): ____ Flow Rates (Initial): ____

(Final): ____ (Final): ____

Total Time: ____ Avg. Flow Rate: ____

Total Volume Sampled: ____

Temperature (°F): ____

BREATH SAMPLING

Meter Readings Final: ____ ft^3 Time of Day: ____

Initial: ____ ft^3 Temperature: ____ °F

Total: ____ ft^3 Duration of Exhalation __min__sec

Correction Factor: X ____ Pump No.: ____

Volume/Cartridge: ____ L (1 ft^3 = 28.3 L)

DRINKING WATER, BLOOD, URINE, MILK SAMPLING

Time of Day: ____ Source (water only) ____
(*e.g.* ground, city, etc.)

Amount Collected: ____

FOOD SAMPLING

Time Period During Which Food was Consumed: ____

Time of Collection: ____

Container	Container Type	Description of Food Type
(1)		
(2)		
(3)		

GENERAL REMARKS

Figure 2. Field Sampling Protocol Sheet (Front)

CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute, Analytical Sciences Division
Chemistry and Life Sciences Group, Research Triangle Park, NC 27709

SAMPLE CODE INAIR 111-5 VO-FB-1	**TENAX BATCH:** 61	**VOLUME COLLECTED:**
SAMPLE TYPE: Tenax	**TEMPERATURE (°F)**	**VOLUME ANALYZED:**

RELIN-QUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	JS	1700	7/11/83	Exposed
JS				

Figure 3. Chain-of-Custody Sheet (Back)

4.4 Once the designated water samples have been collected, enter the time, date, and "Sample Exposed" on the COC and SP sheets for the QC set.

CAUTION

Do not open the QC set bottles for any reason at any time since such exposure may not be representative of sampling conditions. Exposure of the water QC set constitutes the transportation to and from the field, storage, and shipment with the collected field samples.

4.5 Return the QC set with the collected field samples and store in the sample storage refrigerator.

4.6 Have the COC sheets quality-assured by another field staff member.

5.0 EXPOSURE OF FIXED SITE AND PERSONAL AIR QC SETS

QC sets for both fixed site and personal air volatiles consist of a field blank (FB), field control permeation system (FC-PS) and field control flash system (GC-FS) on Tenax cartridges.

5.1 Remove the assigned QC set from the Tenax storage area and place into the field collection can with the culture tubes for the designated participant's samples.

5.1.1 In the event either FC cartridge from the assigned QC set is damaged, lost, or destroyed, disregard the entire set. Assign the next-numbered QC set in its place and repeat sets 3.3-3.5 and 4.1.2 (if applicable).

5.1.2 In the event the FB cartridge from the assigned QC set is damaged, lost, or destroyed, two courses of action are possible.

5.1.2.1 If another unexposed cartridge exists from the same Tenax batch as the FC cartridges, replace the impaired FB cartridge with one from the same Tenax batch and use the label from the impaired cartridge.

5.1.2.2 If no unexposed cartridge from the same Tenax batch as the FB exists, disregard the entire set. Assign the next-numbered QC set in its place and repeat steps 3.3-3.5 and 4.1.2 (if applicable).

5.2 Transport the field collection can to the designated sample collection site.

NOTE: Generally, a single field collection can contains the sampler for all the participants visited during the same sampling period. Therefore, the QC set may be transported to more than one specific sampling site before being returned to storage.

5.3 Once the designated samples have been collected, enter the time, date, and "Sample Exposed," on the QC set's COC sheets.

CAUTION

Do not open any of the QC set culture tubes at any time since such exposure may not be representative of sampling conditions. Exposure of the set constitutes the transportation to and from the field, storage, and shipment with the collected field samples.

5.4 Return the QC set and the collected field samples to the Tenax storage area.

5.5 Store the exposed QC set in a can containing the designated field samples.

NOTE: In order to facilitate the final QA check and packing of the Tenax cartridges prior to shipment, the collected field samples and their corresponding exposed QC sets should be stored in numbered cans as they are collected. As a result, the cartridges are grouped in chronological order and by sets.

5.6 Have the COC sheets quality assured by another field staff member.

6.0 EXPOSURE AND COLLECTION OF BREATH QC SAMPLES

QC sets for breath volatiles consist of a field blank (FB), spirometer blank (SB), field control permeation system (FC-PS), and a field control flash system (FC-FS). Exposure of the two FC and the FB samples is identical to that described in Section 5. However, the SB involves the collection of purified air in the spirometer system just prior to sampling the designated breath sample.

6.1 Remove the assigned QC set from the Tenax storage area and place into the field collection can with the culture tubes for the samples scheduled for collection.

  6.1.1 In the event either FC cartridge from the assigned QC set is damaged, lost, or destroyed, disregard the entire set. Assign the next-numbered QC set in its place and perform steps 3.3-3.5 and 4.1.2 (if applicable).

  6.1.2 In the event either the FB or SB is damaged, lost or destroyed, perform step 5.1.2.

6.2 Transport the field collection can to the participant sampling site.

6.3 Refer to RTI/ACS-SOP-337-001, Collection of Breath Samples (Sections 4.1.7-4.2) and prepare the spirometer accordingly.

  6.3.1 Ready the spirometer as described, installing a clean exhale bag if one has not been previously installed.

6.4 Refer to Figure 4.

  6.4.1 Close ball valve C.

  6.4.2 Close plug valves A and B.

6.5 Disconnect the Teflon air line from plug valve B, insert it into a special mouthbit containing a 1/4 inch opening.

6.6 Insert the mouthbit into the mouthpiece and tighten.

6.7 Open plug valve E, ball valve C, then carefully turn on the air supply.

CAUTION

Do not turn on the air supply before plug valve E and ball valve C are open. The inline back pressure may force the stopper from the humidifier, contaminating the air within, and possibly destroying the humidifier.

6.8 Fill the exhale bag with humidified air. Air should flow easily through the mouthpiece.

6.9 While the exhale bag is being filled, install the SB cartridge on the apparatus in the same manner a field sample cartridge is installed. See RTI/ACS-SOP-337-001, Collection of Breath Samples (Sections 4.3.2.1-4.3.2.4).

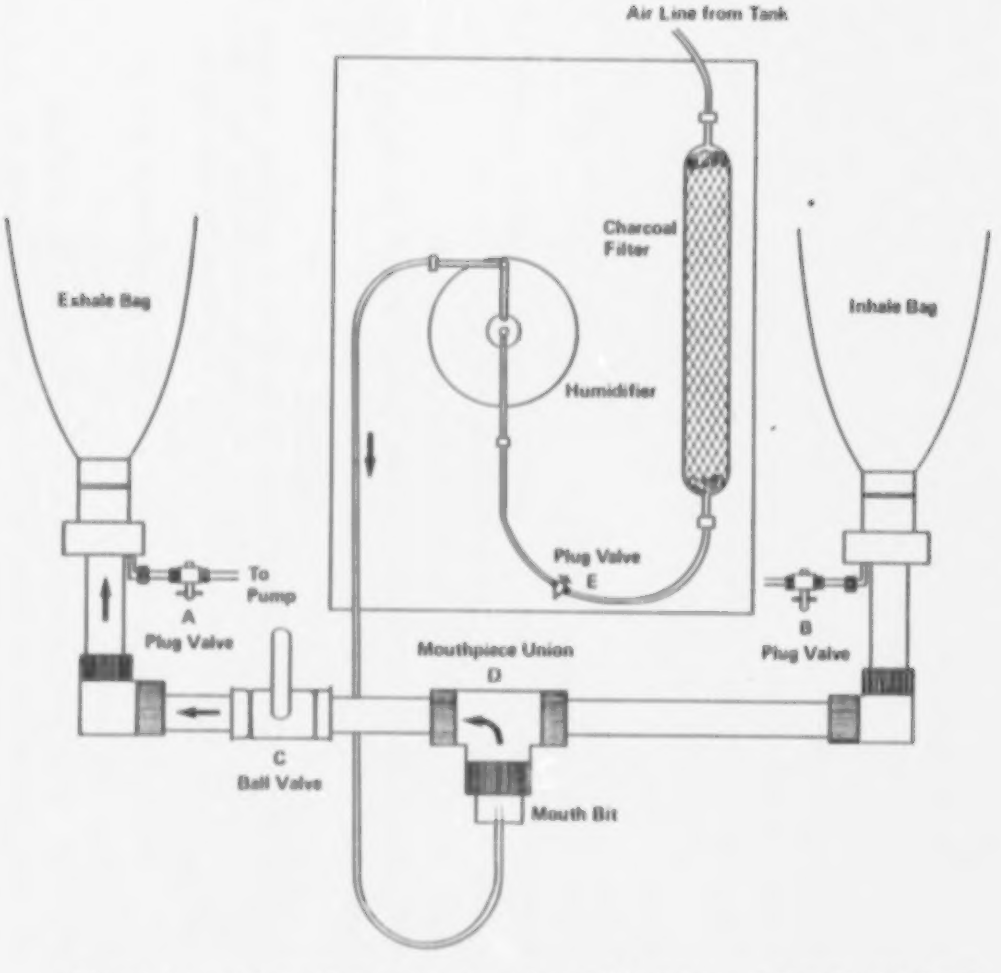
Air Line from Tank
Charcoal Filter
Exhale Bag
Humidifier
Inhale Bag
Plug Valve E
To Pump
A Plug Valve
Mouthpiece Union D
B Plug Valve
C Ball Valve
Mouth Bit

Figure 4. Spirometer Apparatus for Collection of Breath QC Blank

6.10 Record the initial dry gas meter reading from the Nutech sampler.

6.11 When the exhale bag is nearly full, turn off the air supply at the regulator and close ball valve C.

6.12 Open plug valve A.

6.13 Turn on both Nutech samplers and collect the sample.

6.14 Record the time, date, and temperature on the SB SP sheet.

6.15 While the sample is being collected, remove the bottom pad of glass wool from the SB culture tube placing it on a clean Kimwipe®.

6.16 Using a 10 mL glass beaker, add 5 mL of nonindicating Drierite to the culture tube.

6.17 Replace the bottom glass wool pad.

6.18 When the exhale bag is nearly empty, carefully press it against the side of the spirometer to help force out the last of the air.

CAUTION

Do not fold the bag in an effort to force out the air. This may break or damage the bag.

6.19 Once the bag is empty, turn off both Nutech samplers.

6.20 Close plug valve A.

6.21 Using a clean Kimwipe®, carefully remove the Tenax cartridge from the Cajon fitting and place it into the culture tube.

6.22 Using forceps, place the top pad of glass wool onto the Tenax cartridge and cap the culture tube.

CAUTION

Do not allow excess glass wool to lie over the top edge of the culture tube. This may prevent the cap and septum from forming a proper seal with the culture tube, possibly resulting in the contamination of the sample during storage.

6.23 Return the SB to the field collection can.

6.24 Record the final dry gas meter reading from the Nutech sampler onto the SB SP sheet.

6.25 Record the time and date on the COC sheets for the FB and both FC samples.

6.26 Return the QC set with the collected field samples to the Tenax storage.

6.27 Refer to Section 5.4.1 for details on storage of exposed Tenax cartridges.

6.28 Have the COC sheets quality-assured by another field staff member.

RTI/ACS-SOP-862-001
April 1984
Revision 1

Standard Operating Procedure for the Preparation of Water Blanks and Controls

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Vice-President, RTI/ACS Date

Standard Operating Procedure for the Preparation of Water Blanks and Controls

RTI/ACS-SOP-862-001

1.0 SCOPE AND APPLICATION

Blank and control water preparations are used to evaluate the effect of sampling conditions and storage procedures on data quality. Controls consist of purified water which has been fortified with target compounds at concentrations mimicking those of actual water samples. The controls allow evaluation of the analytical procedure and are used to determine losses of target compounds during sampling and storage. Blanks consist of purified water alone. They allow evaluation of sample contamination during storage and sampling. All controls and blanks are prepared in containers identical to those employed in collection of drinking water samples. Once prepared, controls and blanks are stored with the samples and are analyzed at the same time and under the same conditions as samples. Controls and blanks are separated into two groups: those which remain in the laboratory and those which travel to and from the field.

2.0 SUMMARY

All controls and blanks are prepared in the same type bottles used for sample collection. The 64 mL (2 oz.) bottles are cleaned (see RTI/ACS-SOP-322-001) and have sodium thiosulfate added as a preservative. Each bottle is prelabelled before control and blank preparation. Contaminant-free water is prepared by bubbling a cryogenically cleaned helium stream through water which is stirred at 90°C for 90 minutes. After cooling, the contaminant-free water is added to each bottle labelled as a blank, the bottles are tightly capped then stored in the refrigerator. The control bottles are filled nearly full with contaminant-free water. An aliquot of a methanolic calibration solution is then delivered under the water surface, the bottle is filled to the top with water, capped, and stored in a refrigerator. Proper documentation must be maintained, including the preparation of chain-of-custody sheets, for each blank and control. All pertinent data must be entered into a laboratory notebook at the time of preparation.

3.0 MATERIALS AND REAGENTS

3.1 Purified water, distilled, Milli-Q or equivalent.
3.2 64 mL (2 oz.) screw cap bottles.
3.3 20 mm screw caps.
3.4 20 mm Teflon cap liners.
3.5 Pre-coded labels.
3.6 Tape.
3.7 Sodium thiosulfate, Fisher or equivalent.
3.8 Water Preparation Apparatus
    3.8.1 Hot plate stirrer.
    3.8.2 Teflon stir bar.
    3.8.3 A one or two liter Erlenmyer flask.
    3.8.4 Helium or nitrogen gas.
    3.8.5 Liquid nitrogen.
    3.8.6 Liquid nitrogen Dewer.
    3.8.7 Copper gas transfer line with coils in Dewer.
    3.8.8 Glass frit, medium porosity.
    3.8.9 Gas flow controlling valve.
    3.8.10 Thermometer, 0° to 100°C.
3.9 Ice.
3.10 10 µl syringe with Teflon tipped plunger.
3.11 5 mL pipet.
3.12 Pipet bulb.
3.13 Methanolic calibration solution (see SOP "Preparation of Purge and Trap Calibration Solutions).
3.14 Chain-of-custody forms.
3.15 Laboratory notebook.
3.16 Refrigerator.

4.0 PROCEDURE

4.1 Preparation of Bottles

4.1.1 Calculate Number of Blanks and Controls

4.1.1.1 Determine the total number of water samples to be collected.

4.1.1.2 Calculate 5% of the total number of samples.

4.1.1.3 To determine the number of bottles needed, multiply the value from 4.1.1.2 by four.

NOTE: There are four types of blanks and controls. Each type should be prepared at numbers which equal 5% of the total number of water samples to be collected.

4.1.2 Obtain clean 64 mL (2 oz.) bottles (RTI/ACS-SOP-322-001).

4.1.3 Add enough sodium thiosulfate solution so that when the water is evaporated 5 mg of sodium thiosulfate remains in the bottle (e.g., 1.0 mL of 0.5% solution).

4.1.4 Evaporate the water introduced as the sodium thiosulfate solution by placing the bottles in an oven at 110°C for at least two hours.

4.1.5 After cooling, cap the bottles with caps containing Teflon liners.

4.2 Preparation of Water

4.2.1 Add Milli-Q, distilled, or charcoal filtered water to a one or two liter Erlenmeyer flask. Fill the flask nearly to capacity.

NOTE: The size of flask to be used depends upon the number of blanks and controls to be prepared.

4.2.2 Add a Teflon-coated stir bar to the flask. Place the flask on a stirrer/hot plate and begin stirring at a medium rate. A deep vortex should not be formed.

4.2.3 Using a clamp, place the thermometer so that the bulb is centered in the water.

4.2.4 Begin heating the water. The temperature should remain at 90°to 95°C.

4.2.5 Attach 1/4" o.d. copper tubing to the regulator of a helium tank or other clean gas source. The tubing should, at one point, have several coils which fit into a one liter dewar.

4.2.6 Attach the glass tubing of a frit assembly to the copper tube.

4.2.7 Turn on purge gas and purge the tubing to clear any water vapor from the line. (The flow should be approximately 40-60 mL/min.)

4.2.8 Fill the Dewer with liquid nitrogen. Allow the line to be purged for an additional two minutes.

4.2.9 Lower the glass frit into the water to a position just above the stir bar.

4.2.10 Purge the water for 90 minutes. Make sure the Dewer remains filled with liquid nitrogen for the entire purge period.

4.2.11 After ninety minutes, remove the glass frit, turn off the gas flow, and turn off the heat.

*4.2.12 Place a metal tray between the flask and stir plate. Fill the tray with ice. Continue to stir the water.

*4.2.13 Reinsert the glass frit in the flask, turn on the gas flow and continue to purge while the water is cooling.

4.2.14 Cool the water until the temperature reaches 5° to 10°C.

4.3 Labelling Bottles

4.3.1 Label all bottles before the water finishes cooling down.

4.3.2 Obtain TEAM labels assigned for blanks and controls for the site to be visited.

4.3.3 With ball point pen, code each label in numerical sequence by proper designation. For example:

Laboratory Blanks:	WV-LB1
Laboratory Controls:	WV-LC1
Field Blanks:	WV-FB1
Field Controls:	WV-FC1

NOTE: For each new study, begin numbering the blanks and controls from one. Each blank and control must have a unique number for that particular study.

4.3.4 Remove the labels from the backing and place on the bottles.

4.3.5 Wrap transparent tape over the labels and completely around the bottles.

4.3.6 Separate the bottles designated as blanks from those designated as controls.

4.4 Preparation of Blanks

4.4.1 While the prepared water is still cold, pour the water into a beaker, then from the beaker into each 64 mL bottle designated as a blank, until each bottle is nearly full.

NOTE: These transfers should be performed in a room which is free of chemical contaminants, particularly solvents.

4.4.2 Using a clean pipet, transfer enough clean water from the flask to each bottle so that a meniscus is formed at the lip of the bottles.

4.4.3 Carefully cap each bottle with a cap containing a Teflon liner. Make sure no headspace is introduced to the bottle.

4.4.4 Place all of water blanks in a refrigerator at 4°C.

4.5 Preparation of Controls

4.5.1 While the contaminant-free water is still cold, first pour it into a beaker, then quickly into each 64 mL bottle designated as a control until each bottle is nearly full.

CAUTION

If the contaminant-free water is fortified while hot, the more volatile components may be lost to the atmosphere.

4.5.2 Using a 10 μL syringe with a Teflon tipped plunger, aliquot 10 μL of secondary standard B into a single bottle. Always make sure there are no bubbles in the syringe. Deliver the standard solution below the surface of the water.

NOTE: See Table 1 for the description of secondary standard B and the resulting concentrations of target compounds in the controls.

4.5.3 Fill the bottle to the lip with contaminant-free water with the pipet.

Table 1. CONCENTRATION OF TEAM TARGET COMPOUNDS IN WATER CONTROLS

Compound	Concentration in Secondary Standard B (ng/mL)	Resulting Concentration (ng/mL) when 64 mL Water Is Fortified with 10 mL Standard B
Vinylidene chloride	9.92	1.55
Chloroform	380	59.4
1,1,1-Trichloroethane	9.92	1.55
Carbon tetrachloride	9.88	1.54
Benzene	10.0	1.56
1,2-Dichloroethane	10.0	1.56
Bromodichloromethane	74.2	11.6
Trichloroethylene	9.92	1.55
Dibromochloromethane	9.80	1.53
Tetrachloroethylene	10.1	1.58
Chlorobenzene	10.0	1.56
Ethylbenzene	10.1	1.58
p-Xylene	10.0	1.56
Styrene	10.0	1.56
Bromoform	9.84	1.54
m-Dichlorobenzene	10.0	1.56

4.5.4 Cap the bottle tightly with a cap containing a Teflon liner and make sure no headspace is left.

4.5.5 Repeat steps 4.5.2 through 4.5.4 for each bottle.

4.5.6 Transfer all of the water controls to a refrigerator at 4°C.

5.0 DOCUMENTATION

Each blank and control must have a chain-of-custody sheet prepared for it. The chain of custody sheet will follow the blank or control throughout its lifetime and will provide a record for the preparation, transfers, and analysis. In addition to the chain-of-custody form, all pertinent information must be entered into the current TEAM purge and trap laboratory notebook. Proper record keeping is vital because data obtained from the analysis of blanks and controls may be used many months after initial preparation.

5.1 Chain-of-Custody Forms

5.1.1 Obtain a blank chain-of-custody (COC) sheet for each blank and control.

5.1.2 Place a TEAM label corresponding to the proper study on each protocol sheet.

5.1.3 Complete each label with the proper designation. The label on the COC sheet should be identical to that of one blank or control. See Figures 1 and 2.

5.1.4 Enter your initials in the first box of the Received column.

5.1.5 Enter the date of preparation.

5.1.6 If the COC sheet is for a blank, enter Blank Prepared under operation performed. See Figure 2.

5.1.7 If the COC sheet is for a control, enter Control Prepared, the amount of standard used for fortification, the standard used, and the volume of water fortified. See Figure 1.

5.1.8 Enter your initials in the second row in the Relinquished By column.

5.1.9 Place the completed COC sheets in a properly labelled notebook.

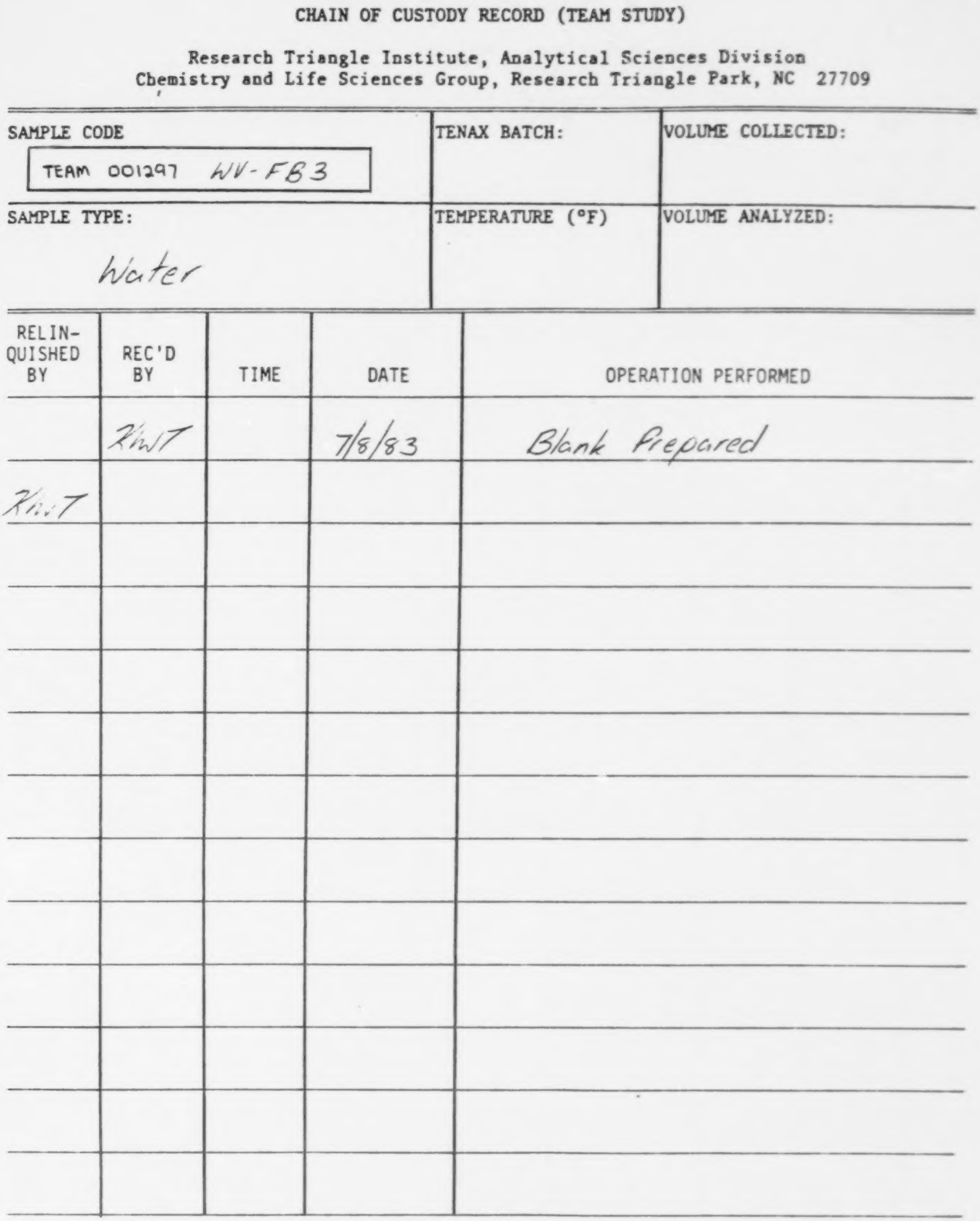
CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute, Analytical Sciences Division
Chemistry and Life Sciences Group, Research Triangle Park, NC 27709

SAMPLE CODE TEAM 001297 WV-FC3	TENAX BATCH:	VOLUME COLLECTED:
SAMPLE TYPE: Water	TEMPERATURE (°F)	VOLUME ANALYZED:

RELINQUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	KWT		7/8/83	Control Prepared 10 uL Std B added to 64 mL water.
KWT				

FIGURE 1. Water Control Chain Of Custody Sheet



CHAIN OF CUSTODY RECORD (TEAM STUDY)

Research Triangle Institute, Analytical Sciences Division
Chemistry and Life Sciences Group, Research Triangle Park, NC 27709

SAMPLE CODE TEAM 001297 WV-FB3	TENAX BATCH:	VOLUME COLLECTED:
SAMPLE TYPE: Water	TEMPERATURE (°F)	VOLUME ANALYZED:

RELIN-QUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	KwT		7/8/83	Blank Prepared
KwT				

FIGURE 2. Water Blank Chain Of Custody Sheet

5.2 Laboratory Notebook

5.2.1 Enter your name and the date of control and blank preparation in the TEAM purge and trap laboratory notebook.

5.2.2 Enter the calibration solution used to fortify the controls.

5.2.3 Enter the volume of calibration solution added to the control bottles.

5.2.4 List the resulting concentration (ng/mL) of each compound in the controls.

5.2.5 List all of the label information for each control and blank prepared.

5.2.6 Explain any changes in the preparation procedure.

RTI/ACS-SOP-881-001 (Air)
SOP-882-001 (Water)
SOP-887-001 (Breath)
December 1983
Revision 0

Standard Operating Procedure for the Submission of Quality Assurance Samples to and Receipt of Data from a QA Laboratory

Author:

Patricia A. Blau
Patricia A. Blau

Approved:

CM Sparacino 12/7/83
Laboratory Manager, RTI/ACS Date

12/5/83
QA Officer, RTI/ACS Date

E Pellizzari 12/5/83
Vice-President, RTI/ACS Date

Standard Operating Procedure for the Submission of Quality Assurance Sample to and Receipt of Data from a QA Laboratory

RTI/ACS-SOP-881-001 (Air)
SOP-882-001 (Water)
SOP-887-001 (Breath)

1.0 SCOPE AND APPLICATION

The intention of this Standard Operating Procedure is two-fold: (1) to document the step-by-step activities required for submission of quality assurance (QA) samples to the QA laboratory and (2) to outline specific instructions for subsequent receipt of results. The methodology herein pertains, specifically, to Tenax cartridges and water samples. It may be applied to other samples and/or studies by modifying the cover letter to the QA lab (and associated paperwork) and the procedure for preparation of samples for shipment (if necessary).

2.0 SUMMARY OF THE METHOD

All QA samples, regardless of matrix, undergo a routine procedure for submission to a designated QA laboratory. This procedure is independent of location of the laboratory and mode of shipment.

Samples are matched to Chain-of-Custody (COC) sheets. A cover letter and list of samples are prepared. Samples are relinquished as noted on the COC sheets. All samples and associated paperwork are carried to the RTI Inventory Control Clerk or Shipping and Supply Specialist.

QA laboratories return the analytical data and signed COC sheets to the originating RTI staff member. The paperwork is filed, and the data are processed.

3.0 PROCEDURE

Three different types of samples are submitted for analysis by a QA laboratory: field samples, exposed field QC samples (check sampling schedule), and QA audit samples (see QA Officer for sample identification numbers).

3.1 Preparation of Samples

Tenax samples for QA analysis are located in a can(s) in the Tenax freezer. The can(s) is coded with a matching code on the corresponding batch of COC photocopies. Water samples for QA analysis are located in numerical order on trays in the TEAM refrigerator.

3.1.1 Tenax Air and Breath Samples

3.1.1.1 Remove the can(s) from the freezer and carry it to the area designated for logging-in activities.

3.1.1.2 Remove the samples from the can(s), keeping inverted culture tubes separate from the rest.

CAUTION

Inverted culture tubes contain nonanalyzable samples and should not be sent to the QA laboratory.

3.1.1.3 Match each sample to a COC sheet.

3.1.1.4 Check to make sure all culture tubes are packed with glass wool between the top of the cartridge and the screw cap so that the Tenax cartridge is immobile when shaken. If any culture tubes lack glass wool, carry them to the "clean room" and insert an unsilanized glass wool plug in each culture tube (using forceps).

CAUTION

Glass wool protruding from the sides of the cap will reduce the effectiveness of the seal and may result in contamination of the Tenax sorbent bed.

3.1.1.5 Return all analyzable QA samples to the can(s).

3.1.1.6 Tightly pack the can(s) with cotton ensuring that each culture tube is surrounded. Place sufficient cotton on top so that the culture tubes do not move when the can is sealed and shaken.

3.1.1.7 Seal the can. Return it to the freezer until the samples are ready for shipment.

3.1.2 Water Samples

3.1.2.1 Locate and separate the samples for QA analysis from the rest of the samples.

3.1.2.2 Match each sample to a COC sheet.

3.1.2.3 Put the samples in any suitable box and return the box to the refrigerator until the samples are ready to be shipped.

3.2 Preparation of Paperwork

3.2.1 Compile a list of codes of QA samples from the COC sheets.

3.2.2 Compose a cover letter. Include all information as shown in the example (Figure 1).

3.2.3 Submit the cover letter and list of samples for typing with a Typing Assignment Form (Figure 2) to the assigned secretary.

3.2.4 Proofread the typed letter and list of samples.

3.2.5 Sign the letter.

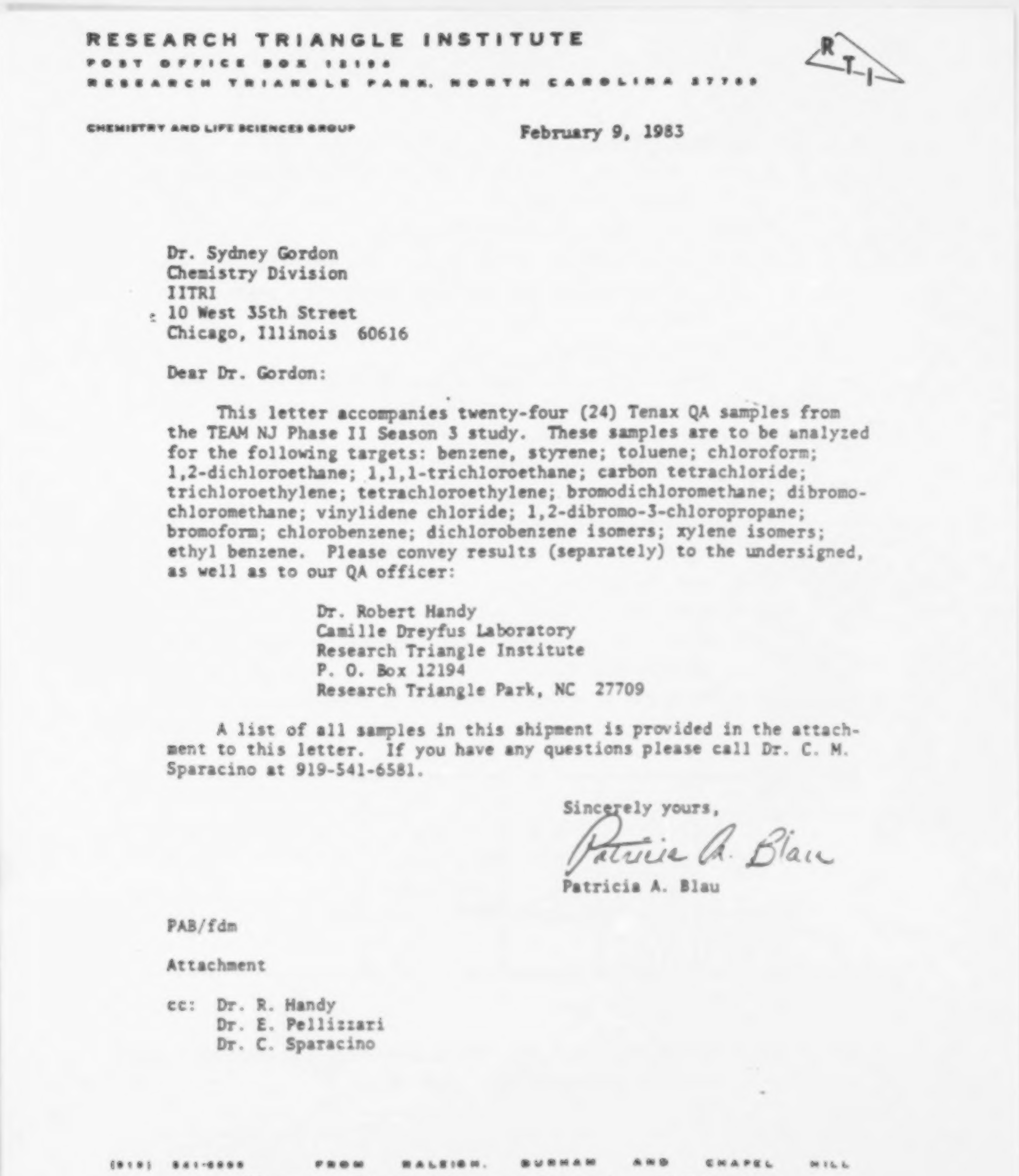
3.2.6 Make four (4) photocopies.

3.2.7 Distribute the copies to the Laboratory Manager, QA Officer and Vice-President of ACS. Keep one for the QA file.

3.2.8 Enter the following information on each COC sheet.

RELINQUISHED BY	REC'D BY	TIME	DATE	OPERATION PERFORMED
	(Your initials)		(Date of intended shipment)	Sent to QA laboratory
(Your initials)				

3.2.9 Attach the letter and list of samples to the COC sheets and put them in an envelope.



RESEARCH TRIANGLE INSTITUTE
POST OFFICE BOX 12194
RESEARCH TRIANGLE PARK, NORTH CAROLINA 27709

CHEMISTRY AND LIFE SCIENCES GROUP

February 9, 1983

Dr. Sydney Gordon
Chemistry Division
IITRI
10 West 35th Street
Chicago, Illinois 60616

Dear Dr. Gordon:

This letter accompanies twenty-four (24) Tenax QA samples from the TEAM NJ Phase II Season 3 study. These samples are to be analyzed for the following targets: benzene, styrene; toluene; chloroform; 1,2-dichloroethane; 1,1,1-trichloroethane; carbon tetrachloride; trichloroethylene; tetrachloroethylene; bromodichloromethane; dibromochloromethane; vinylidene chloride; 1,2-dibromo-3-chloropropane; bromoform; chlorobenzene; dichlorobenzene isomers; xylene isomers; ethyl benzene. Please convey results (separately) to the undersigned, as well as to our QA officer:

Dr. Robert Handy
Camille Dreyfus Laboratory
Research Triangle Institute
P. O. Box 12194
Research Triangle Park, NC 27709

A list of all samples in this shipment is provided in the attachment to this letter. If you have any questions please call Dr. C. M. Sparacino at 919-541-6581.

Sincerely yours,

Patricia A. Blau

Patricia A. Blau

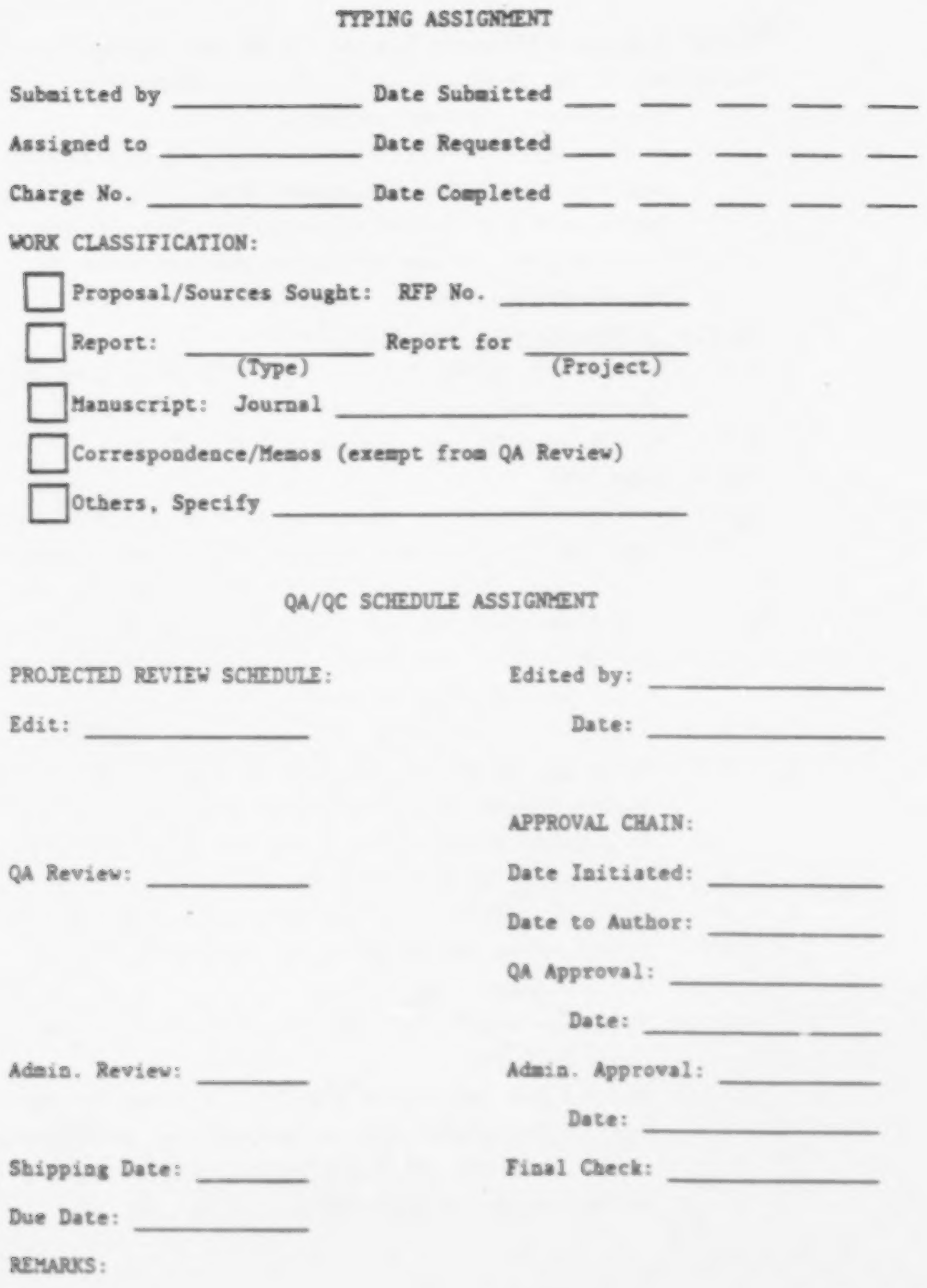
PAB/fdm

Attachment

cc: Dr. R. Handy
Dr. E. Pellizzari
Dr. C. Sparacino

(919) 541-6000 FROM RALEIGH, DURHAM AND CHAPEL HILL

Figure 1. Example of a cover letter to a QA laboratory.



TYPING ASSIGNMENT

Submitted by ____________ Date Submitted ___ ___ ___ ___ ___

Assigned to ____________ Date Requested ___ ___ ___ ___ ___

Charge No. ____________ Date Completed ___ ___ ___ ___ ___

WORK CLASSIFICATION:

☐ Proposal/Sources Sought: RFP No. ____________

☐ Report: ____________ (Type) Report for ____________ (Project)

☐ Manuscript: Journal ____________

☐ Correspondence/Memos (exempt from QA Review)

☐ Others, Specify ____________

QA/QC SCHEDULE ASSIGNMENT

PROJECTED REVIEW SCHEDULE:

Edit: ____________

Edited by: ____________

Date: ____________

APPROVAL CHAIN:

QA Review: ____________

Date Initiated: ____________

Date to Author: ____________

QA Approval: ____________

Date: ____________

Admin. Review: ____________

Admin. Approval: ____________

Date: ____________

Shipping Date: ____________

Final Check: ____________

Due Date: ____________

REMARKS:

Figure 2. Typing assignment form.

3.2.10 Complete a Shipment Clearance Sheet (see Figure 3).

3.2.11 Notify the Inventory Control Clerk or Shipping and Supply Specialist of intended shipment.

3.2.12 Remove samples from storage and carry samples, envelope with paperwork, and Shipment Clearance Sheet to either person listed in 3.2.11 for subsequent packing and shipment. For water samples, include two frozen blue-ice packs (or equivalent) per shipping container.

3.3 Receipt of Results from the QA Laboratory

3.3.1 Make a photocopy of the cover letter received from the QA laboratory and give it to the Laboratory Manager.

3.3.2 Put the original cover letter in the QA file.

3.3.3 Tenax Data

3.3.3.1 Obtain the quantity (in ng) of each analyte spiked onto the control cartridges from the staff member responsible for loading standards (refer to the COC sheet).

3.3.3.2 Enter the field control sample data from the QA laboratory on the QA Data Worksheet for Volatiles (herein referred to as the Worksheet) (Figure 4).

3.3.3.3 Average the control data for each target compound.

3.3.3.4 Enter the background data from the field blank samples analyzed by the QA laboratory on the Worksheet.

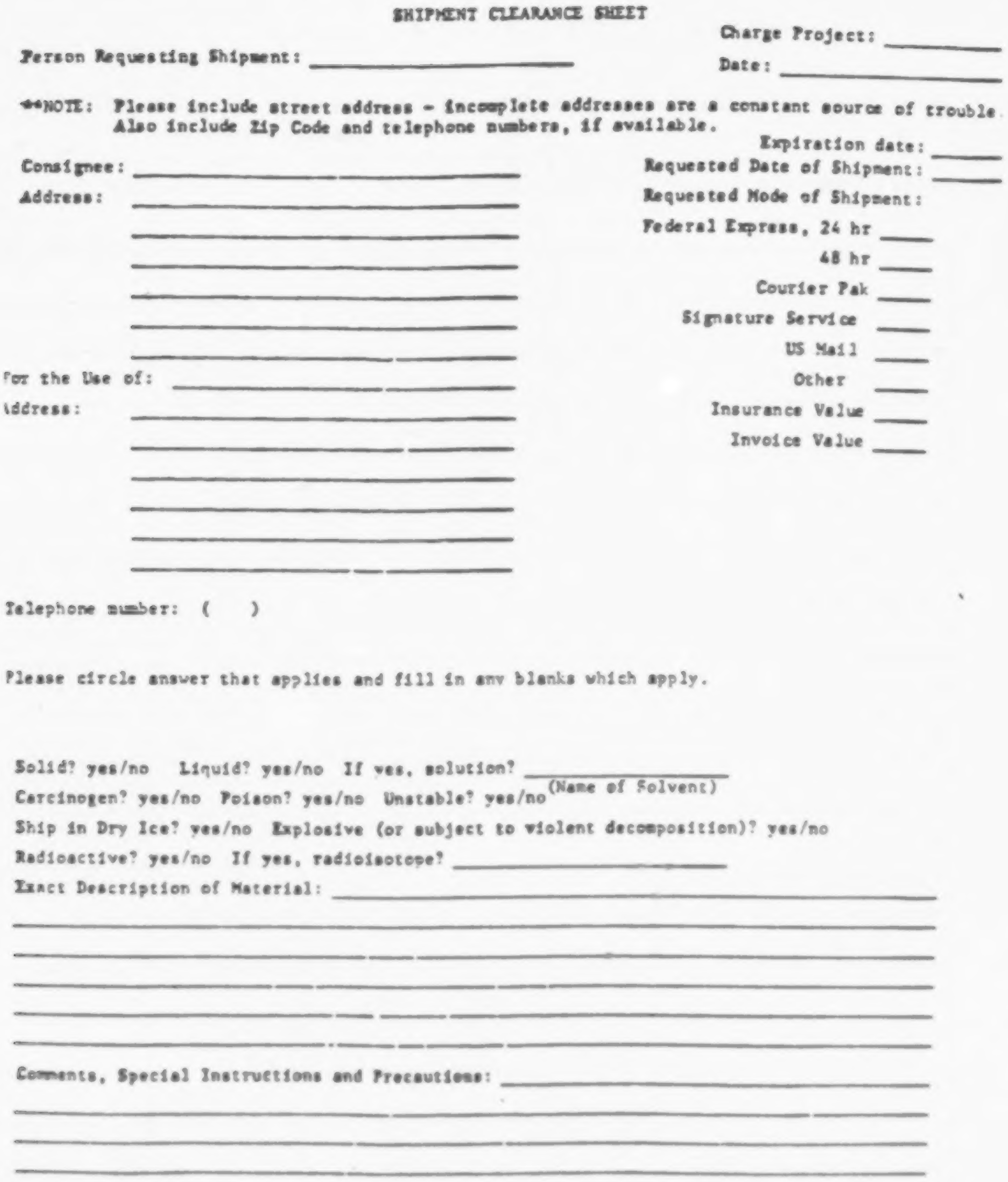
3.3.3.5 Average the background data for each target compound.

3.3.3.6 Subtract the averaged background (if any) from the averaged control values for each target compound.

3.3.3.7 Divide by the amount spiked to determine % recovery for each target compound.

3.3.3.8 Make a photocopy of the QA laboratory data and the Worksheet.

3.3.3.9 Submit these photocopies plus the COC sheets to the staff member responsible for quantitation of TEAM target compounds in air and breath Tenax samples (using the HP2000 computer program "Quant 6").



SHIPMENT CLEARANCE SHEET

Charge Project: ________

Person Requesting Shipment: ____________________

Date: ____________

**NOTE: Please include street address - incomplete addresses are a constant source of trouble.
Also include Zip Code and telephone numbers, if available.

Expiration date: ____

Requested Date of Shipment: ____

Consignee: ____________________

Address: ____________________

____

____

____

____

____

Requested Mode of Shipment:

Federal Express, 24 hr ____

48 hr ____

Courier Pak ____

Signature Service ____

US Mail ____

Other ____

Insurance Value ____

Invoice Value ____

For the Use of: ____________________

Address: ____________________

____

____

____

____

____

Telephone number: (   )

Please circle answer that applies and fill in any blanks which apply.

Solid? yes/no Liquid? yes/no If yes, solution? ______________
(Name of Solvent)

Carcinogen? yes/no Poison? yes/no Unstable? yes/no

Ship in Dry Ice? yes/no Explosive (or subject to violent decomposition)? yes/no

Radioactive? yes/no If yes, radioisotope? ______________

Exact Description of Material: ______________________________

____

____

____

____

____

Comments, Special Instructions and Precautions: ______________________________

____

____

____

Figure 3. Shipment clearance sheet.

QA Data Worksheet - Part A

Matrix: ______________ Trip: __________________ Units: Air/Breath--ng
Water--ppb

Target Compound	Quantity Reported							
	Field Control Code				Field Blank Code			
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								

Figure 4. QA data worksheet - Part A.

QA Data Worksheet - Part B

Matrix: ______________ Trip: ________________ Units: Air/Breath--ng
Water--ppb

Target Compound	Quantity Reported from Part A Field Control Mean	Field Blank Mean	Corrected Control	Amount Spike	Percent Recovery
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					

Figure 4 (Cont'd.). QA Data Worksheet - Part B.

Code Number	Target Compound
1	Vinylidene chloride
2	Chloroform
3	1,2-Dichloroethane
4	1,1,1-Trichloroethane
5	Benzene
6	Carbontetrachloride
7	Trichloroethylene
8	Bromodichloromethane
9	Dibromochloromethane
10	Toluene
11	Tetrachloroethylene
12	Chlorobenzene
13	Bromoform
14	Dibromochloropropane
15	Styrene
16	m-Dichlorobenzene
17	p-Dichlorobenzene
18	o-Dichlorobenzene
19	Ethylbenzene
20	o-xylene
21	m-xylene
22	p-xylene

Figure 4 (Cont'd.). QA Data Worksheet - Part C.

3.3.3.10 Return the original QA laboratory data and Worksheet to the QA file.

3.3.4 Water Data

3.3.4.1 Obtain the quantity (in ppb) of each volatile spiked into the water control samples from the staff member who spiked them (refer to the COC sheet).

3.3.4.2 Follow procedures 3.3.3.2 through 3.3.3.7 above for the water data.

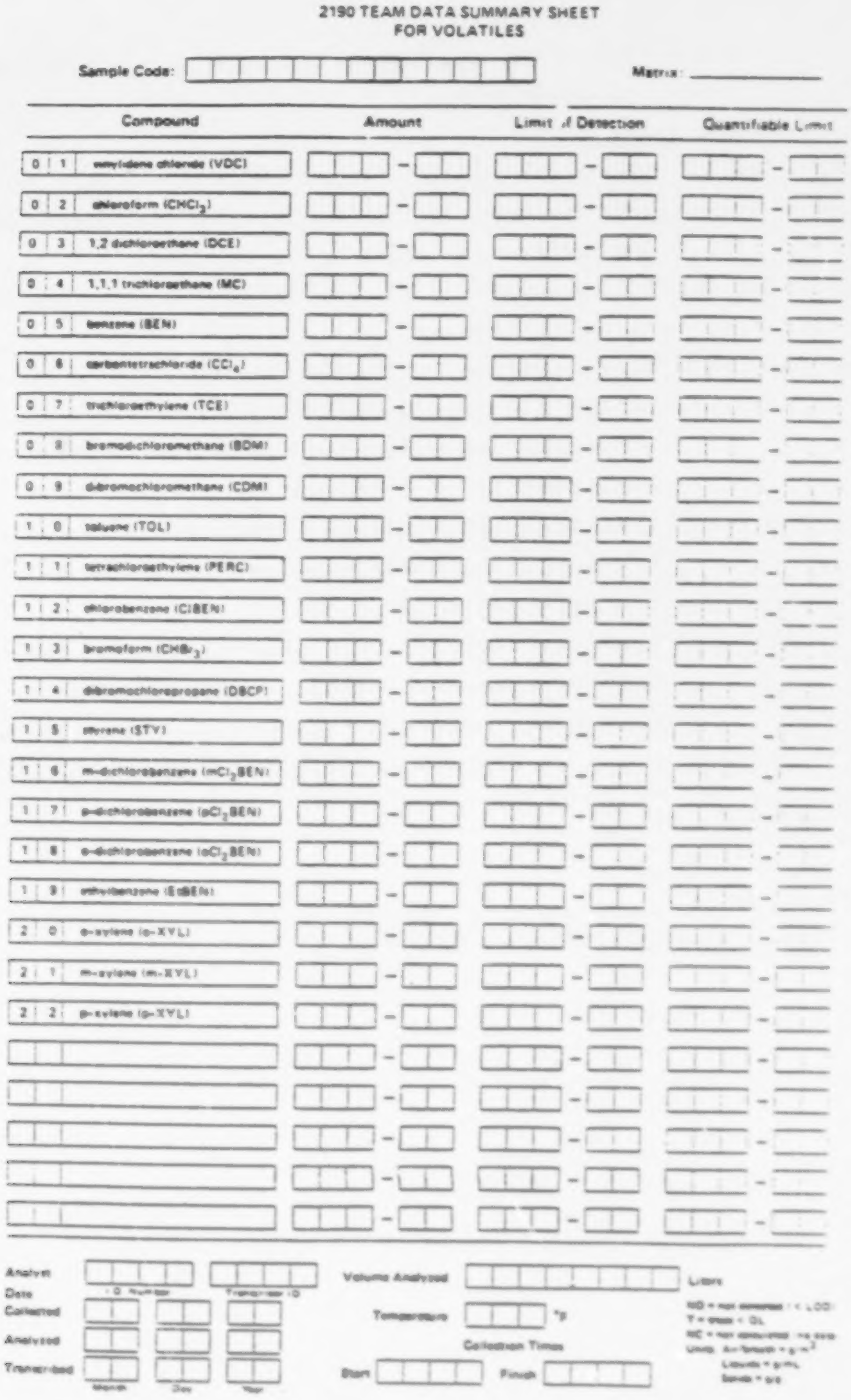
3.3.4.3 Make a photocopy of the QA laboratory data and Worksheet.

3.3.4.4 Correct all field sample data for recovery on the photocopy. Enter the corrected value next to the uncorrected value in red ink.

3.3.4.5 Complete a TEAM Data Summary Sheet for Volatiles (Figure 5) for each field sample analyzed by the QA laboratory (excluding blanks and controls).

3.3.4.6 Submit the completed Data Summary Sheets, a copy of the QA Data Worksheet for Volatiles and COC sheets to the staff member responsible for QA review and batching.

3.3.4.7 Return the original QA laboratory data and Worksheet to the QA file.



2190 TEAM DATA SUMMARY SHEET
FOR VOLATILES

Sample Code: ☐☐☐☐☐☐☐☐☐☐☐☐☐ Matrix: ____________

Compound	Amount	Limit of Detection	Quantifiable Limit
0 1 vinylidene chloride (VDC)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
0 2 chloroform ($CHCl_3$)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
0 3 1,2 dichloroethane (DCE)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
0 4 1,1,1 trichloroethane (MC)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
0 5 benzene (BEN)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
0 6 carbontetrachloride (CCl_4)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
0 7 trichloroethylene (TCE)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
0 8 bromodichloromethane (BDM)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
0 9 dibromochloromethane (CDM)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
1 0 toluene (TOL)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
1 1 tetrachloroethylene (PERC)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
1 2 chlorobenzene (ClBEN)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
1 3 bromoform ($CHBr_3$)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
1 4 dibromochloropropane (DBCP)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
1 5 styrene (STY)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
1 6 m-dichlorobenzene (mCl_2BEN)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
1 7 p-dichlorobenzene (pCl_2BEN)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
1 8 o-dichlorobenzene (oCl_2BEN)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
1 9 ethylbenzene (EtBEN)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
2 0 o-xylene (o-XYL)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
2 1 m-xylene (m-XYL)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
2 2 p-xylene (p-XYL)	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐
☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐	☐☐☐ - ☐☐

Analyst ☐☐☐☐ ☐☐☐☐
I.D. Number Transcriber I.D.

Volume Analyzed ☐☐☐☐☐☐☐☐ Liters

Date	Month	Day	Year
Collected	☐☐	☐☐	☐☐
Analyzed	☐☐	☐☐	☐☐
Transcribed	☐☐	☐☐	☐☐

Temperature ☐☐☐ °F

Collection Times
Start ☐☐☐☐ Finish ☐☐☐☐

Figure 5. TEAM data summary sheet for volatiles.

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END